COMPARISON OF TENSILE BOND STRENGTH OF TWO DIFFERENT RESIN CEMENTS USED TO BOND BASE METAL ALLOY TO HUMAN ENAMEL -AN INVITRO STUDY

Dissertation Submitted to

THE TAMILNADU DR. M.G.R. MEDICAL UNIVERSITY

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CERTIFICATE

This is to certify that this dissertation title **"COMPARISON OF TENSILE BOND STRENGTH OF TWO DIFFERENT RESIN CEMENTS USED TO BOND BASE METAL ALLOY TO HUMAN ENAMEL-AN INVITRO STUDY"** is a bonafide record of work done under our guidance during the study period between 2004-2007.

This Dissertation is submitted to THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY, in partial fulfillment for the Degree of **MASTER OF DENTAL SURGERY- PROSTHODONTICS, BRANCH VI.** It has not been submitted (partial or full) for the award of any other degree or diploma.

RAGAS DENTAL COLLEGE Chennai

GUIDE

Dr. N.S.AZAGHARASAN, M.D.S
Professor & HOD,
Department of Prosthodontics
Ragas Dental College & Hospital
Chennai.

PROFESSOR & HEAD
DEPT OF PROSTHODONTICS
Ragas Dental College & Hospital
Chennai - 600 113.

Dr.S.RAMACHANDRAN, M.D.S
Principal
Ragas Dental College & Hospital
Chennai.

PRINCIPAL
RAGAS DENTAL COLLEGE AND HOSPITAL
UTHANDI, CHENNAI-600 119.

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Introduction

INTRODUCTION

Base metal alloys were introduced into dentistry by Erdle RW and Prange CH[5] in 1930s. These alloys are based on more than 75% of base metal elements. They have been of immense value in dentistry because of their low cost and their influence on weight, strength, stiffness, improved ceramic bonding and corrosion resistance[5]. They are used in metal ceramic restorations, all metal restorations, bonded restorations and removable partial dentures.

The bonding mechanism between the cast restoration and the tooth structure can be mechanical, chemical or a combination of the two. Mechanical retention is achieved through sealing irregular crevices along both the tooth and the metal surfaces by cement. The bond strength in such a situation depends on the strength of the luting agent. Chemical bonding is achieved by using aqueous cements based on polyacrylic acids through chelation of acrylic acids to both organic and inorganic components of teeth. Resin-based cements using some speciality functional groups also have exhibited chemical bonding[4].

The fixed prosthesis can de-bond because of biological factors, physical factors, or a combination of the two. The disintegration of the cement due to fracture or erosion leads to plaque accumulation and secondary caries and hence biologic failure of the restoration results[42,55]. The introduction of newer cements with low solubility, high strength and fluoride releasing abilities overcomes the biologic failure of restoration[59, 55].

The physical factors like intraoral forces, film thickness and flaws within the cement layer also influence the quality of the bond. The use of cements with high tensile strength values, thin film thickness and/or a bond enhancing intermediate layer to maximize the effect of inherent strength on the restoration, can enhance the bond strength of the cement. The resin cements have been developed to fulfill these requirements. The recently published literature suggests the use of resin cements for obtaining optimum retention for indirect restorations[25, 49].

The success of bonding of cast restoration to the human enamel with resin cements depends upon optimization of the following components[38,4],

1) The enamel-to-resin bond. 2) The cohesive bond of the composite resin.

3) Resin-to-framework bond.

The enamel-to-resin bond is micromechanical in nature. It depends upon proper etching and bonding procedures. Though resin cements have been found to provide optimum retention, they may cause postoperative sensitivity in some cases. In order to reduce post-operative tooth sensitivity, it has been recommended that an adequate self-etching primer be employed before the restoration is cemented. Studies have suggested that the self-etching primer helps to improve the tensile bond strength of cement. Many such systems are currently being employed[16].

However, the luting procedure with those cements are carried out in two stages, initially by application of self-etching primer and followed by the application of the cement itself. In order to reduce the operator variability and the chair side time, resin cement with self-etching primer incorporated into it has been introduced recently and the cementation procedure can be carried out in a single stage [14]. These cements have been termed as self-adhesive universal resin cements. However studies with regard to the performance of this latter type of cement are limited [49].

The cohesive bond of the cement contributes to improved bonding of alloy to the tooth surface. Cohesive failure can occur through the cement layer itself, when chemical bonding is involved. Failure can also occur along the interfaces (cement-tooth interface and cement-prosthesis interface), if the bonding is mechanical.[4]

The resin to framework bond is essential for successful restoration. It has been reported that the bonding between the metal and resin is purely mechanical. Attempts have been made to increase the bond strength by surface treatment of the metal with methods such as silicoating[13, 35], acid etching [36,38,39], air abrasion [7,34,56], use of bonding agents [28,7], ultra sonic cleaning [56], acid soak[34]and electrolytic etching[61, 57]. Among these methods, air abrasion has proved to be a simple yet adequate method of improving the resin-metal interface bonding[34].

Various laboratory studies have been done on layer thickness[3], adherence energy[6], polymerization shrinkage[60], and the effect of storage conditions[1] of cast restoration on the bond strength of resin cements. The bond strength of the resin luting cements used to bond the base metal alloy to the tooth structure is an important feature that must be investigated. Comparisons among different studies are complicated because of the different approaches used to test adhesive ability (bonding) of resin cements. Generally, adhesive capacity has been evaluated with invitro testing, with shear and tensile tests. However, finite element analysis[10] concluded that shear test were the most efficient to disclose the cohesive resistance of the material, whereas tensile tests were better to investigate the adhesion at the interface. Since the purpose of this study was to evaluate the adhesive capacity of the resin cement rather than the stress produced during clinical function, a tensile test was used.

In light of the above, the aim of this study was

1. To compare the tensile bond strength of two different resin cements used to bond base metal alloy to human enamel.
2. To evaluate and compare the bond strength of two different resin cement used after surface treatment of the base metal alloy with two grades of air abrasive to bond base metal alloy to human enamel.
3. To study the type of bond failure by scanning electron microscope by examination of the debonded surface.

Review of literature

Laufer B-z et al (1973) [35] this study tested the tensile bond strength of two commercial resin luting cements to a variety of metal alloys coated with SiO_x -C and compared these values to an acid Ni-Cr alloy. He concluded that coating the metal alloys with Silicoat material resulted in a composite bond strength that was approximately twice that of etched Litecast B alloy and the use of intermediate unfilled resin is necessary to obtain a consistently high bond strength between the resin cement and metal.

Moser J. B et al (1973) [42] studied the tensile bond strength between three polycarboxylate cements and Types I and IIIgold, 304 stainless steel and cobalt-chrome alloy was compared to that obtained with a silico-phosphate and a zincphosphate cement. The polycarboxylate cements showed higher strengths with almost all alloys than did the other two.

Piwowarczyk A et al (1973) [49] this study determined the shear-bond strength of cementing agents to high-gold-content alloy castings and different dental ceramic. it was concluded that after 14 days of water storage followed by thermal cycling, only the self-adhesive universal resin cement and 2of the resin cements exhibited strong bond strengths of specific prosthodontic materials.

Rochette A.L (1973) [51] introduced a technique for union between gold alloy splint and enamel. This report described a technique for fabricating a splint on mandibular anterior teeth without tooth reduction. Applying a coupling agent to the gold and etching the enamel to enhance attachment with sevitron attained the fixation.

Nicholls J.I (1974) [44] studied the crown retention by the effect of convergence angle variation on the computed stresses in the luting agent. He conclude that a tensile failure in the luting agent appears to be the most likely and then in order to evaluate a clinical situation, both load magnitude are equally important and must be given special attention if an accurate determination of induced stress is required

Hoard et al (1978) [31] investigated the role, duration, and the magnitude of the intra coronal pressures developed during the seating of full crowns. A moral system was developed which recorded the intra coronal pressure during crown cementation at three locations simultaneously. Peak pressures and residual pressures were greatest with zinc phosphate cement. It was small with zinc oxide and intermediate with poly carboxylate cement. The uneven intra coronal pressure in the cement suggests a complex flow pattern capable of developing the separation of phases.

Brauer G.M et al (1979) [12] studied the strength and durability of the dentin-acrylic resin cemented with 2-cynoacrylate esters. Maximum adhesion was obtained with iso-butyl 2-cynoacrylate after 1% acid pretreatment of the dentin. Hydrolytic stability was improved by addition of polymer to the adhesive or coating around the joint.

Coelho et al (1986) [19] studied the effect of surface treatment on nickel chromium alloy and its effect on tensile bond strength of resinous cement. The following surface treatment was done 1) air abrasion with 50µm aluminum oxide, 2) air abrasion with 50mm glass beads, 3) air abrasion with a mixture of aluminum oxide and glass beads (ratio 1:1), 4) air abrasion with aluminum oxide and immersion in acid solution of potassium permanganate, and 5) air abrasion with aluminum oxide and immersion in potassium solution of potassium permanganate. No statistically significant result was obtained among the experimental groups.

Creugers N.H.J et al (1986) [22] compared three types of resin-retained cast metal prosthesis. The three retainer designs were 1) perforated metal framework with five holes of 1mm diameter covering the lingual surfaces of the abutment teeth, 2) perforated metal framework with five holes of 1mm diameter, covering the lingual surfaces and part of the proximal surfaces adjacent to the edentulous area, and 3) Etched metal framework with one non retentive venting hole covering the lingual surface. They concluded that micromechanical retainers were found to be more retentive than macromechanical retainers.

Hill G.L et al (1986) [30] investigated the effect of errors in estimating the surface area on the bond strength of eight base metal alloy used for etched, cast metal; resin-bonded technique. The results were 1) under estimation of surface area and under etching of berilium Ni-Cr alloys will have less negative effect than over estimation and over etching. 2) Over estimation of surface area and over etching a nonberilium Ni-Cr alloy do not reduce the bond strength.

Livaditis j (1986) [38] introduced a method for chemically etching a selected non-noble alloy to create micromechanical retention of resin-bonded retainers. This report provided data supporting that an effective attachment is created between resins and etched metal. This system reduces the disadvantage in creating micromechanical retention by the electro chemical approach.

Felton D.N et al (1987) [27] investigated the effect of surface roughness of crown preparation and retention of cemented castings. The purpose of this investigation was to compare the retention of crowns cemented on teeth prepared with carbide burs with crown cemented on teeth prepared with diamond burs. They concluded that teeth prepared for full crowns for using diamond burs will have 31% greater retention than preparations made with carbide bur.

Ferrari M et al (1987) [28] studied on the possibility of an effective bond between air abraded retainers and etched enamel retainer with the adhesive composite and investigated into the microscopic appearance of the materials at the interfaces. This study was primarily concerned with visual observation of the bonded surface of the luting agent with the etched enamel and abraded metal. Microscopic examination offers a favorable method of observing the relationship of a luting material to etched enamel and to the abraded surface of retainer.

Watanabe F (1988) [61] the objective of this study was to determine in vitro tensile bond strength of three adhesive cements and two resin bonded bridge cements to alloys each with two surface preparations. Sandblasted Ni-Cr-Br alloy, electro-etched Ni-Cr-Be alloy, sandblasted Type IV gold, and tin-plated Type IV gold alloy. Storage conditions of 24 hours at 37°C and 30 days at 70°C were evaluated. The adhesive cements usually failed cohesively under these conditions, where as the resin-bonded bridge failed adhesively at the cement alloy interface.

Tanaka et al (1988)[57] introduced a new Ion-coating treatment of alloys for dental adhesive resins. Ion-coating the surface of the alloys resulted in strong bonds with adhesive resins, and after 100,000 thermocycles, bond strength of above 20 MPa was maintained.

Atta OM et al (1990) [7] in this study sandblasted surfaces of beryllium-free, nickel-chromium alloy were bonded with one of three chemical adhesives. After thermal cycling the bonded specimens were tested for shear and tensile strength. He concluded that panavia Ex material produced the strongest tensile and shear bonds when adhered to sandblasted nickel-chromium alloy. These bond strength did not change thermal cycling.

Kohli S et al (1990)[34] studied the effect of three different metal surface treatment on the tensile strength of the resin bond to non-noble nickel-chromium-beryllium alloy by bonding metal to metal. The metal surfaces was subjected to one of following treatments and bonded: (1)etched chemically with Assure-Etch etchant and bonded with Comspan Opaque cement,(2)etched chemically with Met-Etch etchant and bonded with Comspan Opaque cement, and (3) air abraded with 50µm alumina particles and bonded with Panavia EX. He concluded that the high tensile strengths obtained with all three groups suggest that they may all be used with success clinically to bond cast restorations and the bond strengths obtained with these systems are all atleast three times higher than the enamel-to-resin bond.

Ishijima et al (1992) [32] Investigated the bond strength of a composite resin bonded to various dental casting alloys with three adhesive systems- Silicoater, Panavia, and Superbond C&B .the metal surfaces were treated with aluminium oxide blasting before application of adhesive. Thermal cycling caused a reduction in bond strength for all combination of adhesive system and alloys, the Silicoater system recorded the greatest bond strength. The 4-META system was equivalent to Panavia system in bond strengths to most metals and exhibited greater strength.

Juntave. N and Millstein PL (1992)[33] studied the effects of varying luting agents and internal surface roughness with different types of cores and cements were studied. This study explored crown retention as it was related to 1) core material, 2) luting agents, 3) thickness of cement, 4) internal surface roughness of castings and 5) effect of thermal stress. They concluded that 1) amalgam was superior to other core materials regardless of other luting agents used 2) zinc phosphate and resin luting agents were more retentive than glass ionomer luting agents 3) thermal cycling reduce the retentive bond strength 4) luting agents film thickness of50 and 100μm were more retentive than a luting agent film thickness 150μm 5) retainers with coarse internal surface than those with smooth internal surfaces.

Mojon.P et al (1992) [41] This study was designed to evaluate the bond strength of glass-ionomer cement to a precious PFM alloy, to determine the influence of cement as it matured, and to compare the results with the bond strength created by zinc phosphate cement and an adhesive resin cement containing 4-META. The results showed that the zinc phosphate cement was the weakest material, whereas the adhesive resins produce the strongest joints.

Tjan A.H.L and Li T (1992) [58] examined the retentive property of cast gold complete crowns cemented with an adhesive resin cement (Panavia Ex) was compared with retention of crowns cemented with zinc phosphate cements (Flecks) and the conventional resin cement (Compsan). The effect of these agents on seating of crowns also was evaluated. Both resin cements provided better seating of crowns than zinc phosphate cement.

White et al (1992) [62] examined the effect of seating force on the film thickness of new adhesive luting agents. The materials tested were zinc phosphate cement, glass ionomer cement, poly carboxylate cement, and resinous cement with a dentinal bonding agent. They concluded that the nature of setting reaction effected film thickness. The faster the cement sets the less time available for flow to achieve optimal film thickness.

Yoshida K et al (1993) [65] studied the effect of three adhesive metal primers on the shear bond strength of a light-cured prosthetic composite resin bonded to cobalt-chromium or silver-palladium-copper-gold casting alloy. Results of this study indicated that the ceased primer, which contained the phosphoric acid monomer MDP, was effective in strongly bonding light-cured veneering resin to Co-Cr alloy.

White N.S et al (1994) [63] studied micro leakage for cast crowns. Standardized tooth preparations were completed on previously intact human molars in vivo, and castings were made with a precious metal ceramic alloy by conventional techniques. The castings were randomly assigned to the following luting agents: zinc phosphate, composite resin-glass ionomer hybrid, and a composite resin-glass ionomer hybrid with a dentinal bonding agent and were cemented in a standardized manner to periodontally compromised molars. After 6 months the teeth were carefully extracted, stained, embedded, and sectioned, and the in vivo microleakage was measured. ANOVA disclosed significant differences between groups, and a multiple comparisons test revealed that the zinc phosphate group leaked significantly more than other cement groups.

Alster D et al (1995) [3] The aim of this study was to determine the tensile strength of resin composite joints cure in restrained conditions between two parallel metal surfaces as a function of resin composite thickness. It was concluded that if adhesion to tooth structure were improved thinner adhesive joints might enhance the clinical success of luted restorations.

Asmussen E et al (1995) [6] This study investigated whether a relationship exist between adherence energy to a metal substrate and the degree of cross linking and wetting characteristics of experimental resin-based luting agents. The measured wetting characteristics were work of adhesion and surface tension, and their dispersive and polar components. Those cements with a low degree of cross-linking and their monomers, which were relatively polar, resulted in high adherence values.

Bona AD and Noort van R (1995) [10] evaluated the shear vs. tensile bond strength of resin composite to bonded to ceramic. The contention of this study was that the shear bond strength test was inappropriate and inadequate for the vitro assessment of resin composite bonded to ceramic. A variety of shear bond strength test arrangements was assessed by finite element analysis .it was concluded that a tensile bond strength measurement technique for ceramic bonded to resin composite as a more suitable alternative.

Attin T et al (1996) [8] studied the influence of enamel conditioning on bond strength of resin modified glass ionomer restorative materials and polyacid-modified composites. This study evaluated enamel bond strength of restorative materials containing both glass ionomer and composite components. Three resin-modified glass ionomer restorative materials, three polyacid-modified composites, a hybrid composite and a chemical-cured glass-ionomer cement were tested for enamel tensile bond strength with and without conditioning of the tooth surfaces. Tensile bond strength was determined for five specimens each of conditioned and unconditioned bovine teeth. No significant difference was observed between the hybrid composite and the tested materials attached with the phosphoric acid etching technique. To improve adhesion of the tested materials to enamel, following the manufacturers' instructions about tooth surface conditioning is recommended. Superior bond strength to enamel was obtained for polyacid-modified composites, which are attached with the phosphoric acid etching technique and thereby resemble the adhesion patterns of composites

Gates W.D et al (1996) [29] compared the tensile bond strengths of two base metal alloys and two noble metal alloys, tin-plated and non-tin-plated, with an adhesive resinous cement. Two tin platers were compared for their effectiveness in enhancing the composite resin-to-metal bond. The following conclusions were drawn from the study,

- The mean tensile bond strength of non-tin –plated noble and high noble alloys was significantly lower than both the tin-plated noble and high noble alloys and the non-tin-plated base metal alloys.
- The mean tensile bond strength of the tin-plated alloys was not significantly different from the tensile bond strengths of base metal alloy.
- The mean tensile bond strength of the samples tin plated with the different tin platers was not significantly different.

Yoshida K et al (1996) [64] compared the durability and shear bond strengths of combinations of three adhesive primers and three resin cements bonded to silver-palladium-copper-gold and cobalt-chromium alloys. The adhesive luting cements Imperva Dual, Panavia21, and SuperbondC&B and the adhesive primers Metal primer material, V-Primer material, and ceased Opaque primer material were used. He concluded that with the combined use of adhesive primer and resin luting cement-to-cement fixed prosthodontic restorations, complicated surface modification of dental casting alloys may be negligible and the crowns and fixed prostheses will be capable of withstanding long-term clinical use.

De Kanter R.J.A.M et al (1998) [24] The purpose of the study was to collect survival data on posterior resin-bonded bridges hich were placed under controlled clinical conditions, and to find possible relationships between 1) survival and the bonding system and 2) survival and the abutment tooth preparation design. It was concluded that preparation of grooves in abutment teeth for posterior resin-bonded bridges are beneficial to their chance of survival. Resin-bonded bridges placed in the maxilla have a better prognosis than those made in mandible. The bonding system used in this study had no influence on the chance of failure.

Rosenstiel S.F et al (1998) [53] reviewed the dental luting agents. This review identified biologic, mechanical, esthetic, and working properties of an ideal material and summarized published information as to how available materials conform to those ideals and how their performance was affected by manipulative variables.

Li C Z and White N .S et al (1999) [37] evaluated the mechanical properties of cement and determined that

- Luting cements differed considerably with respect to mechanical properties
- Storage time influenced elastic moduli and it affected various cements
- Resin-composite and resin-modified glass ionomer cements displayed lower elastic moduli than other types of cement
- Cements exhibited different failure mechanism in compression
- Tenderness toward strain rate sensitivity were found
- Cements dominated by resinous components exhibited markedly tougher behavior in flexure than other cements

El-Mowafy O (2001) [25] the use of resin cements in restorative dentistry to overcome retention problems. He concluded that excessive attachment might make it impossible to remove crowns and fixed partial dentures so the use of carefully selected resin cements in conjunction with reliable bonding agents can help.

Lopes et al (2002) [40] discussed some aspects of dental adhesion, and its importance. A recently introduced adhesive technique was described in an effort to compare their principal advantages and existing difficulties. They concluded that self-etching primer systems have undergone a rapid evolution over the past few years and the evolution of adhesive systems has resulted in bond strengths to dentin are very close to that of enamel.

Pontes D.G et al (2002) [50] compared the microleakage of new all-in-one adhesive systems on enamel and dentin margins with that of a conventional total-etch system. Thirty buccal class V cavities were prepared in enamel and dentin margins and randomly divided into three groups of ten specimens each. Group I was bonded with Etch and Prime, group II with Prompt L-Pop, and group III with 35% phosphoric acid plus Prime and Bond 2.1. Among the three adhesive systems used in the study, Prompt L-Pop provided the least microleakage in enamel and there was no significant difference among the groups on dentin margins.

Christensen G J et al (2003) [15] This article discusses the subject of retention and what can be done to improve it in the long term with crowns and fixed prostheses. He suggested resin cements than the conventional cements for providing additional strength when inadequate retention is suspected.

Mota.C.S et al (2003) [43] The purpose of the study was to evaluate the tensile bond strength of 4 resin luting agents to bovine enamel and dentin. It was concluded that the tensile bond strengths of resin luting agents to enamel were higher than those to dentin.

Bouillaguet S (2004) [11] reported biological risks of resin-based materials to the dentin-pulp complex. The clinical success of new restorative techniques has been attributed to the ability of resin-based material to seal resin-tooth interface in the absence of any adverse biological effect. The formation of a perfect seal around resin-based restorations was further required to offer an effective protection to the dentin-pulp complex against microbiological risks.

Christensen G J et al (2004) [14] this article discusses the various types of mechanical retentive methods available and make suggestions about the use of bonding agents for achieving optimum retention for restorations. Increased use of titanium alloy or pure titanium pins and judicious use of potholes, channels, undercuts, grooves and box forms can yield good results.

Bishara S.E et al (2005) [9] compared the shear bond strength of two self-etch primer/adhesive systems on unprepared enamel and orthodontic brackets. The brackets were bonded to human enamel by 1) a two step self-etch primer/adhesive system was used and 2) a one-step self-etch, self-adhesive resin cement was used. They concluded that by reducing the number of steps during bonding, clinicians were able to save time as reduce the potential for error and contamination during the bonding procedure.

Cotert H S et al (2006) [20] observed the effect of alloy type, resin type, and simulated loading on enamel-resin-metal tensile bond strength. The following were the conclusions

- Alloys used in the study did not reveal any significant difference in enamel-resin-metal bond strength.

- Cyclic loading had a weakening effect on enamel-resin-metal bond strength.
- Interactions calculated between the variables of this study were insignificant.

Materials and Methods

The following materials were taken up in the study to compare tensile bond strength of two different resin cements used to bond base metal to human enamel after the surface treatment with two grades of air abrasion.

1. MATERIALS

1. 40 freshly extracted non carious permanent human incisors. (Fig.1)
2. Self-Cure Acrylic resin (DPI-RR Cold Cure, Dental Products India Ltd, Mumbai, INDIA). (Fig.2)
3. Base metal alloy:
    - Nickel Chromium Alloy (Heraenium S, Heraeus Kulzer Gmbh, Gruner Weg, Hanau, GERMANY). (Fig .3)
4. Investment material:
    - Phosphate bonded investment (Moldavest Exact, HeraeusKulzer, Gmbh, Gruner Weg, Hanau, GERMANY). (Fig .4)
5. Colloidal silica –
    - Investment BS Liquid 1 (Heraeus Kulzer, Gmbh, Gruner Weg, Hanau, GERMANY). (Fig.5)
6. Aluminum oxide – A. 50 microns (Delta., INDIA) (Fig. 6)

   B. 250 microns (Protechno, SPAIN) (Fig.7)
7. Pattern resin (GC corporation, Tokyo, JAPAN) (Fig.8)
8. Resin cements employed to compare tensile bond strength were

a. Panavia F.2 (Kuraray medical inc. JAPAN) (Fig.9)

b. RelyX Unicem (3M,ESPE GERMANY) (Fig.10)

9. Custom made wells of diameter 5mm and 1mm depth on a metal block. (Fig.11)
10. Sprue wax, 2.5mm green-wax wires for sprues, (Bego, GERMANY) (Fig.12)
11. Paper liner (Heraus paper ring liner, Heraeus Kulzer GmbH, GERMANY). (Fig .13)
12. Surfactant spray (George Taub products, USA)(Fig.14)
13. Alloy casting rings of 4cm diameter and 5cm length. (Fig .15)

2. INSTRUMENTS AND EQUIPMENTS:

- Lab Micromotor (Strong series, Saeshin Precision Find.Co. KOREA) (Fig.16)
- Grit silicon carbide paper (180,240,320, 400,600).
- Visible light cure unit (3M ESPE, GERMANY)(Fig.17)
- High frequency mixing Amalgamator (Gnatus Amalga mix II, Gnatus Equipmentos Medico-Odentologicos LTD, BRAZIL)(Fig.18)
- Sand blaster (Renfert, GERMANY) (Fig.19)

- Induction casting machine (Fornax GEU, Bego, GERMANY) (Fig.20)
- Ultrasonic cleaner. (KOREA)(Fig.21)
- Thermocycling unit - custom made. (Fig.22)

3. TESTING EQUIPMENTS:

- Universal Testing Machine (Instron, GERMANY) (Fig .23)
- Scanning electron microscope (Leo stereoscan 440,Oxford,LONDON) (Fig.24)

Methodology

For testing the tensile bond strength of the two different resin cement system bonded between base metal alloy and human enamel the following methodology was followed as shown in flow chart below.

40 natural teeth embedded in self-cure acrylic with their labial surface exposed

40 pattern resin discs were made from custom-made metal block

40 pattern resin discs were relined on the enamel surface with pattern resin for adaptation

Investing and casting procedures carried out and they were randomly divided into four groups

Group 1

10 samples

Group 2

10 samples

Group 3

10 samples

Group 4

10 samples

Surface treatment with 50μ Al_2O_3

Surface treatment with 250μ Al_2O_3

Surface treatment with 50μ Al_2O_3

Surface treatment with 250μ Al_2O_3

Luting with Panavia F.2 cement

Luting with RelyX Unicem cement

Thermocycling

Tensile Bond Strength testing (Instron testing machine)

Debonded alloy surface examined with scanning electron microscope

Results

Statistical Analysis

A. Preparation of the natural teeth specimens:

Forty, non-carious, extracted human central incisors of comparable crown sizes were used in the study. They were stored in normal saline till the study had commenced. (Fig.1)

B. Preparation of the natural tooth test specimens:

The crown portion of the teeth were cut from their roots under water cooling and were then embedded in self-curing acrylic resin blocks measuring 12mm × 12mm × 30mm with their labial surfaces exposed (Fig.25). Each specimen was marked for future reference. The labial surfaces of the test teeth were ground at slow speed, with an 180grit silicon carbide paper mounted on a slow speed hand piece to create a flat surface of enamel. After this process the teeth were visually examined to ensure that the enamel layer was continuous and the dentin was not exposed. Only teeth, which conformed to the above, were taken for the study and remaining were discarded. In this way, 40 natural tooth specimens embedded in self cure acrylic resin blocks were obtained

C. Preparation of metal discs:

A custom made metal block with circular wells of 5mm diameter and 1 mm depth was used for making discs using pattern resin (GC corporation, Tokyo, JAPAN)(Fig.26). Each disc was adapted to the natural tooth surface test specimen and it was relined again with pattern resin (Fig.8) to ensure close adaptation to the corresponding enamel surface (Fig.27). A U-shaped wax (Bego, Germany) loop was placed on each disc to serve as a sprue during investing and casting (Fig.28). Later, the same loop served as the attachment to be connected to the universal testing machine (Instron, Germany) (Fig.23). The sprue former with reservoir was attached to U-shaped loop (Fig.29). The completed pattern with the sprue former was attached to the crucible former.

Investing procedure was carried out using phosphate bonded investment material (Moldavest Exact, HeraeusKulzer, Gmbh, Gruner Weg, Hanau, Germany) (Fig.4) according to manufacturer's instructions. After wax elimination (burnout) procedure, the casting was done in an induction-casting machine (Fornax GEU, Bego, GERMANY)(Fig.20). The Nickel-Chromium alloy (Heraenium S, Heraeus Kulzer Gmbh, Gruner Weg, Hanau, GERMANY) (Fig.12), was heated sufficiently (melting point 1260-1350°C casting temperature-1500°C) till the alloy ingot turned to molten state, and the crucible was released and centrifugal force ensured the completion of the casting procedure. Investment was allowed to cool down to room temperature. The casting procedure followed was same for all the forty test samples. Following casting, the hot casting ring was bench cooled to room temperature, and then the cylinder of investment containing the casting was pressed out from the ring. The investment cylinder was cleaved along its long axis, and the casting was lifted free. Sandblasting using macro abrasive aluminum oxide divested the casting. The sprue was removed with an ultra thin abrasive disc. The cast discs were steam cleansed and checked visually. The surface of the casting was inspected and finishing procedures was done. Each disc was also labeled and stored in individual boxes along with the tooth samples for future identification. Thus each disc of metal was customized to have a close adaptation to its corresponding natural tooth specimen. In this way, 40 metal discs of corresponding natural tooth were obtained. These discs were randomly divided into four groups. Groups 1 and 3 were surface treated with 50 μm aluminum oxide before luting similarly Groups 2 and 4 were surface treated with 250 μm aluminum oxide before luting.

The four test groups are detailed below:

Group no	Resin Cement used for bonding	Aluminum oxide abrasive particle size in µm
1	Panavia F.2	50
2	Panavia F.2	250
3	RelyX Unicem	50
4	RelyX Unicem	250

The bonding procedure employed for obtaining the samples for each of the above test group is detailed below

Group 1

In this group ten corresponding cast metal disc were air abraded with 50µ aluminum oxide particles and were cleaned in an ultrasonic cleaner before bonding to tooth enamel mounted on acrylic block with Panavia F.2 (Kuraray medical Inc, JAPAN) (fig.9) resin cement. The cementation procedure according to manufacturer’s instruction is as follows, One drop of ED primer II (Kuraray medical Inc, JAPAN) liquid A and liquid B was dispensed into the well of mixing dish and mixed. It was then applied on the tooth surface and left for 30 seconds (Fig.34) Equal amount of paste A and paste B was mixed on the mixing plate for 20 seconds (Fig.35a) and then the cement was applied on the metal disc (Fig.35b) and placed on the enamel surface (Fig.36). It was then light cured for 20 seconds (Fig.37).

Group 2

In this group ten corresponding cast metal disc were air abraded with 250µ aluminum oxide particles and were cleaned in an ultrasonic cleaner before bonding to tooth enamel mounted on acrylic block with Panavia F.2 (Kuraray medical Inc, JAPAN) (fig.9) resin cement. The metal discs were cemented to the corresponding tooth specimen using the same procedure as followed for group 1 specimens.

Group 3

In this group ten corresponding cast metal disc were air abraded with 50µ aluminum oxide particles and were cleaned in an ultrasonic cleaner before bonding to tooth enamel mounted on acrylic block with RelyX Unicem (3M ESPE.GERMANY) (Fig.10) resin cement. The cementation procedure according to manufacturer's instruction is as follows, the resin capsule was inserted into the Applicap Activator (3M ESPE. GERMANY) (Fig.38) and the activator lever was pushed down and held for 2 seconds. RelyX Unicem (3M ESPE, GERMANY) Self-Adhesive Universal Resin was mixed using high-frequency mixing amalgamator (Gnatus Amalga mix II, Gnatus Equipmentos Medico-Odentologicos LTD, BRAZIL)(Fig.18) for 15 seconds. Longer mixing causes minimal acceleration of setting. Shorter mixing should be avoided. After completion of the mixing, the capsule was inserted into the applicator and the nozzle was opened as far as possible (Fig.39). The cement was applied on the metal disc and then placed on the enamel surface. It was then light cured for 20 seconds (Fig.37).

Group 4

In this group ten corresponding cast metal disc were air abraded with 50µ aluminum oxide particles and were cleaned in an ultrasonic cleaner before bonding to tooth enamel mounted on acrylic block with RelyX Unicem (3M ESPE.GERMANY) (Fig.10) resin cement. The metal discs were cemented to the corresponding tooth specimen using the same procedure as followed for group 3 specimens.

D. Thermocycling Procedure:

Thermocycling in vitro is a common way of testing dental materials to aid in establishing suitability for in vivo use .In this study, all the 40 test specimens were subjected to thermocycling. The specimens were stored in distilled water for 1 week during which they were thermocycled. Thermocycling unit was custom fabricated. It consists of thermocouple and a heating element. A temperature sensor kept in the water bath was connected to a digital display unit. The digital display unit had a set button through which a temperature could be accurately set to ± 1°C. When the water bath attains the desired temperature the thermocouple automatically cuts off the power supply and there by maintains the set temperature. Temperature of 8°C - 55°C was set with this unit. The temperature of 8°C was maintained with ice pack containing crushed ice and the temperature was measured through the thermometer. The specimens were thermocycled between 8° C and 55°C water bath for 500 cycles with a 20sec dwell time and 10-second transfer time. Then the specimens were ready for testing.

E. Preparation of Testing Hook

A custom made testing hook was required to engage the U-shaped loop of the test specimen in the instron testing machine for tensile testing. Testing hook was cast using base metal alloy and were then embedded in self curing acrylic resin blocks measuring 20mm x 20mm x 20mm. (Fig.40)

F. Testing Procedure:

Each acrylic resin block was mounted firmly to the lower jaw of the universal testing machine (Instron, GERMANY) (Fig.23) at the Department of Composite Technology, Indian Institute of Technology, Chennai, INDIA. The test configuration was then loaded in tension, with the generation of tensile forces perpendicular to the etched-metal interface at a cross head speed of 0.5mm/min until failure occurred. The failure loads were recorded. The tensile bond strength was calculated by dividing the failure load by the surface area of the bonding area.

The debonded surfaces of alloys were examined with scanning electron microscope (Leo stereoscan 440, Oxford, LONDON,)

(Fig.24) at Crystal Growth Department, Anna University, Chennai, INDIA.

G. Evaluation of tensile Bond Strength

The breaking load values were recorded through a computer connected to Instron testing machine. The values obtained were in ‘Kg’ and bond strength was calculated in MPa using the formula mentioned below.

Newton (N) = Kg x 9.81

$$\text{Bond strength (MPa)} = \frac{\text{Load (N)}}{\text{Surface Area (mm}^2\text{)}}$$

H. Statistical analysis

The SPSS software package was used for statistical analysis (SPSS for Windows 8.0, SPSS Software Copr, Munich, Germany). Mean and standard deviation were estimated from the sample for each study group. Mean values were compared by student’s t-test. Equality of variances was studied by Levene’s test .In the present study, $P < 0.05$ was considered as the level of significance.

Figures

FIGURES

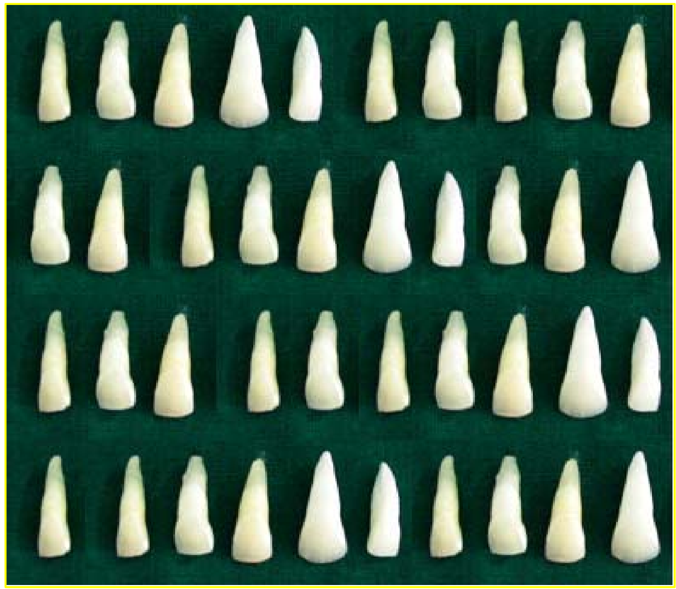


Fig 1 - Forty extracted teeth

Fig 2 - Self cure acrylic resin

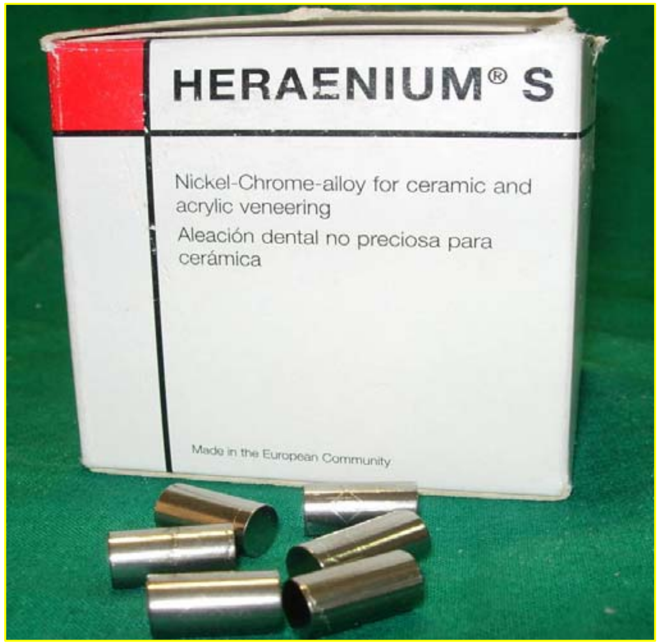


Fig 3 - Base metal alloy

Fig 4 - Phosphate bonded investment material

Fig 5 - Colloidal silica

Fig 6 - Aluminum oxide 50 microns

Fig 7 - Aluminum oxide 250 microns

Fig 8 - Pattern resin

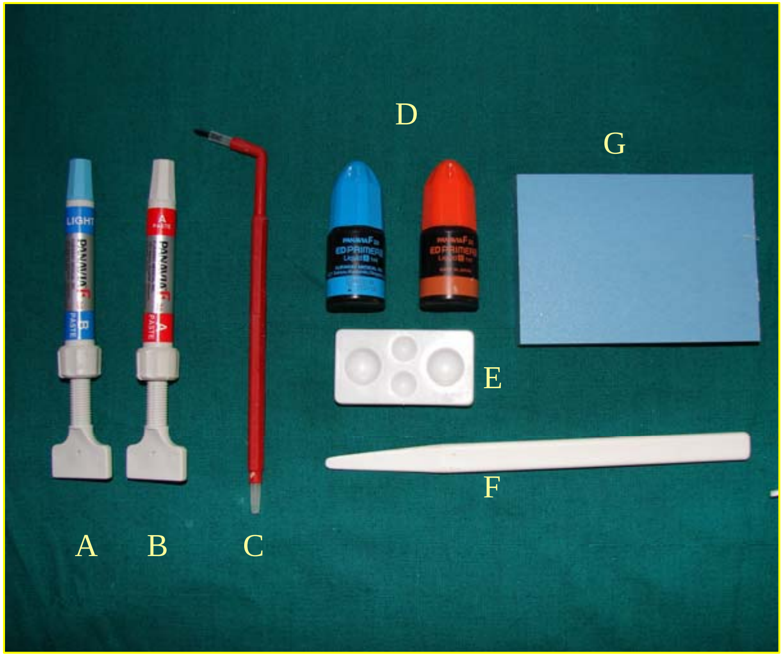


Fig 9 - Panavia F.2 resin cement system

A - Paste A B- Paste B C- Small brush holder with brush

D - ED Primer II (liquid A&B) E - Mixing dish F - Spatula
G – Mixing Pad

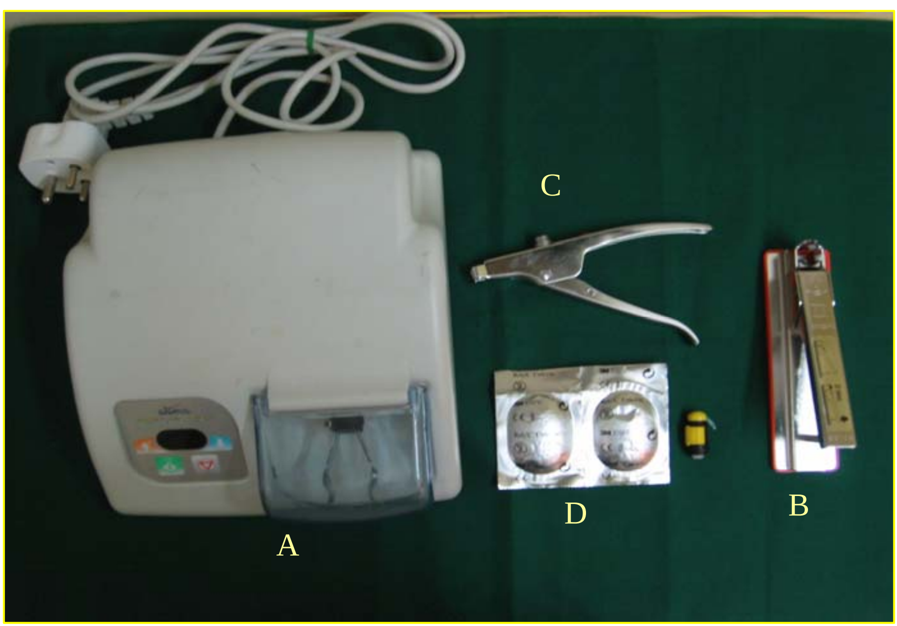


Fig 10 - RelyX Unicem resin cement system
A – High frequency mixing amalgamator B – Applicap activator

C – Applicator D – Cement capsule

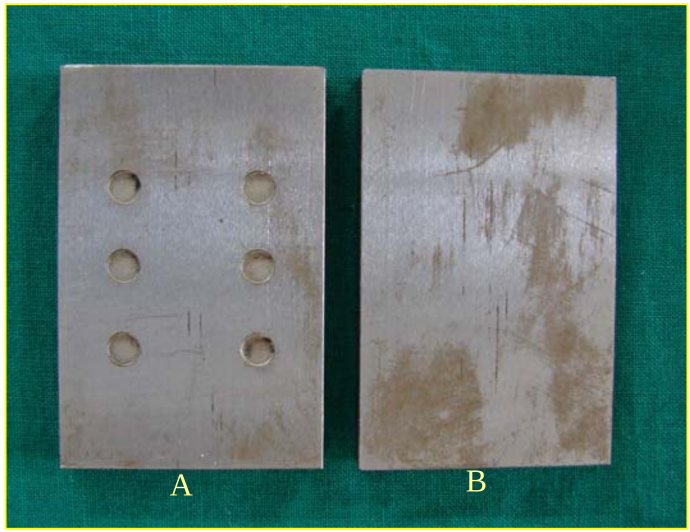


Fig 11 - Custom made metal block with wells (A) and its counter (B)

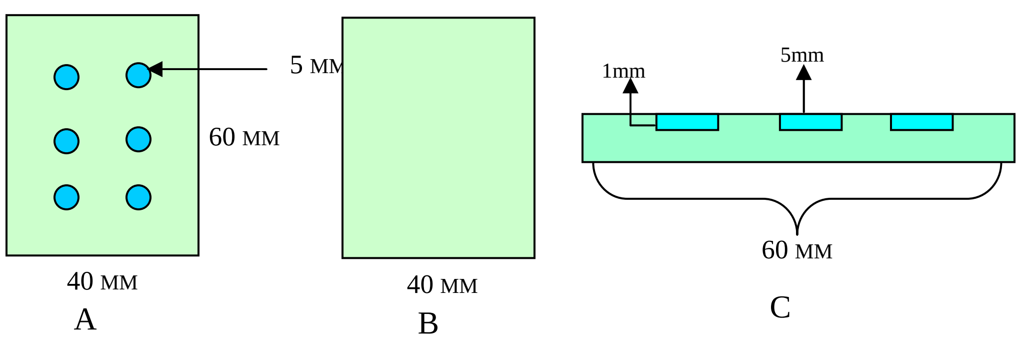


Fig 11a – Line diagram of custom made metal blocks with wells (A) and its counter (B)
Side view cross section (C)

Fig 12 - Sprue wax 2.5mm

Fig 13 - Ring liner

Fig 14 - Surfactant spray

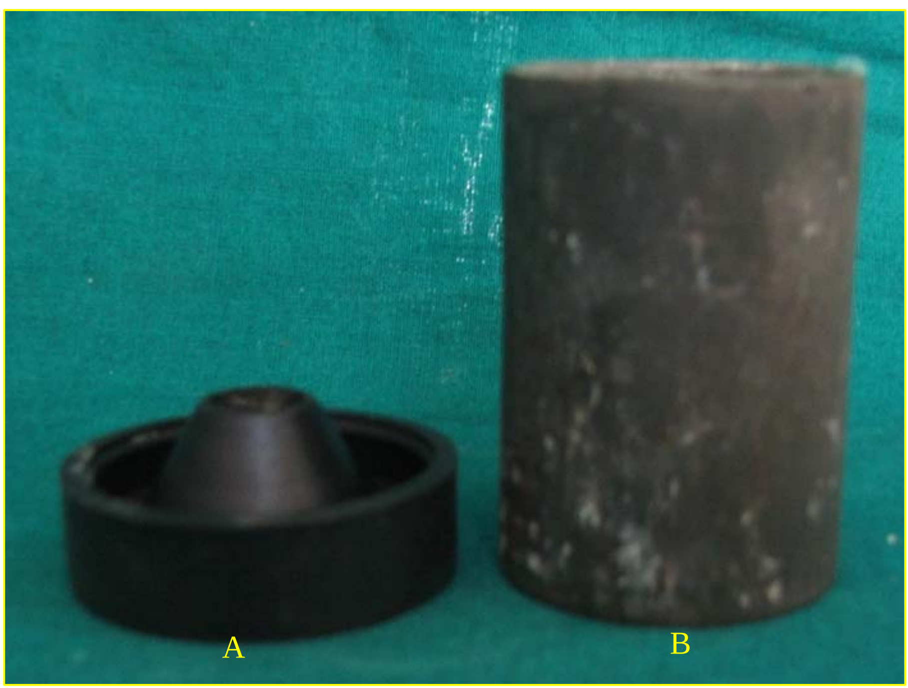


Fig 15 - Crucible former (A) and Alloy casting ring (B)

Fig 16- Lab Micromotor

Fig 17 – Visible light cure unit

Fig 18 – High frequency mixing amalgamator

Fig 19 – Sand blaster

Fig.20.Induction casting machine

Fig.21.Ultrasonic cleaner

Fig 22 - Thermocycling unit

Fig 23 - Instron testing machine

Fig 24 - Scanning electron microscope

Preparation of natural tooth specimen

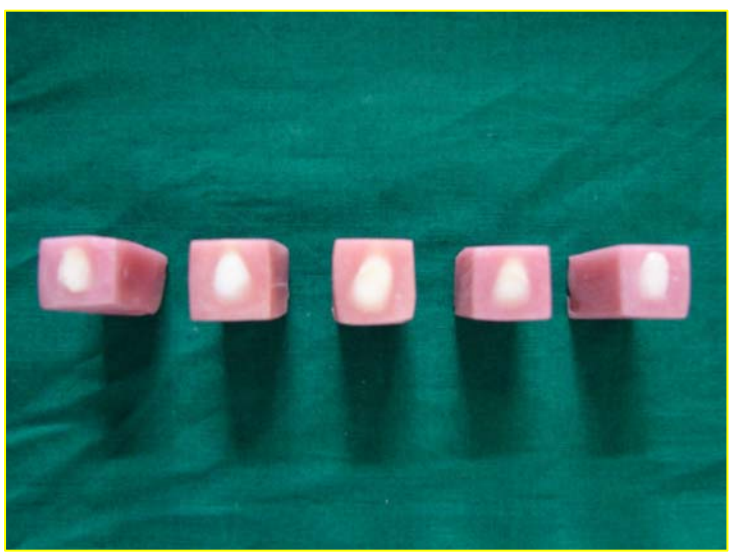


Fig 25 -Prepared crown of incisor tooth portion embedded in self-cure acrylic resin blocks

PREPARATION OF METAL DISCS

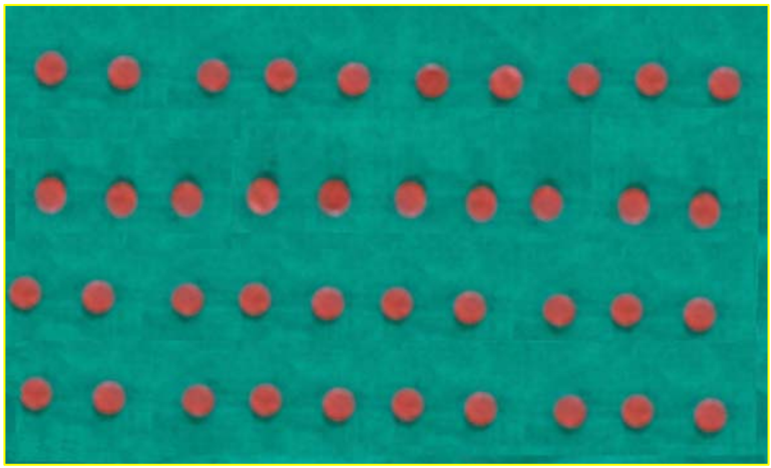


Fig 26 - Pattern resin discs

Fig 27 - Relining of pattern resin disc over prepared tooth sample

Fig 28 U shaped loop attached to pattern resin disc

Fig 29 Completed pattern and sprue attached to Crucible Former

Fig.30 Pattern in position in the casting ring

Fig.31 Divested casting

Fig.32 Sandblasted casting

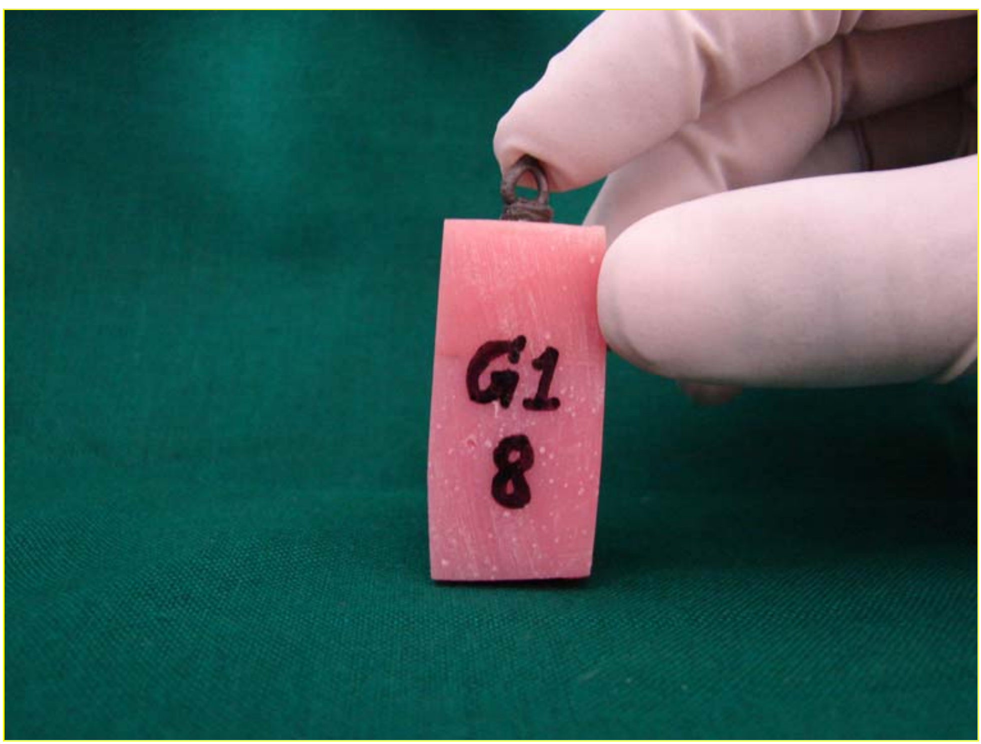


Fig.33 Try in of cast metal disc on tooth specimen

Cementation procedures

Fig.34 Application of E D primer on tooth specimen for Panavia resin system

Fig.35A Mixing of paste A and paste B of Panavia resin system

Fig.35B Panavia resin cement applied on the metal surface

Fig.36 Cementation of metal disc to the tooth specimen

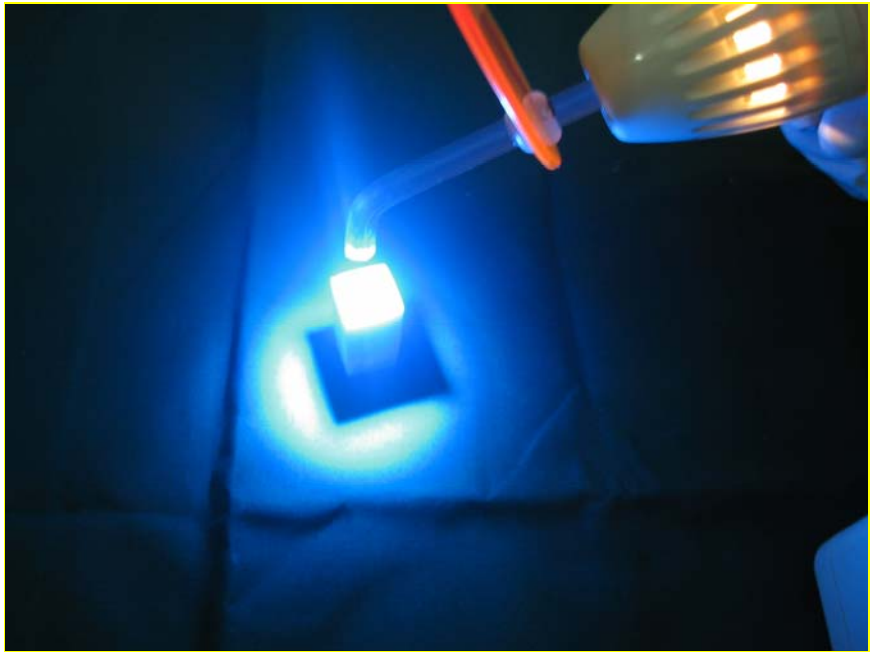


Fig.37 light curing of the resin cement

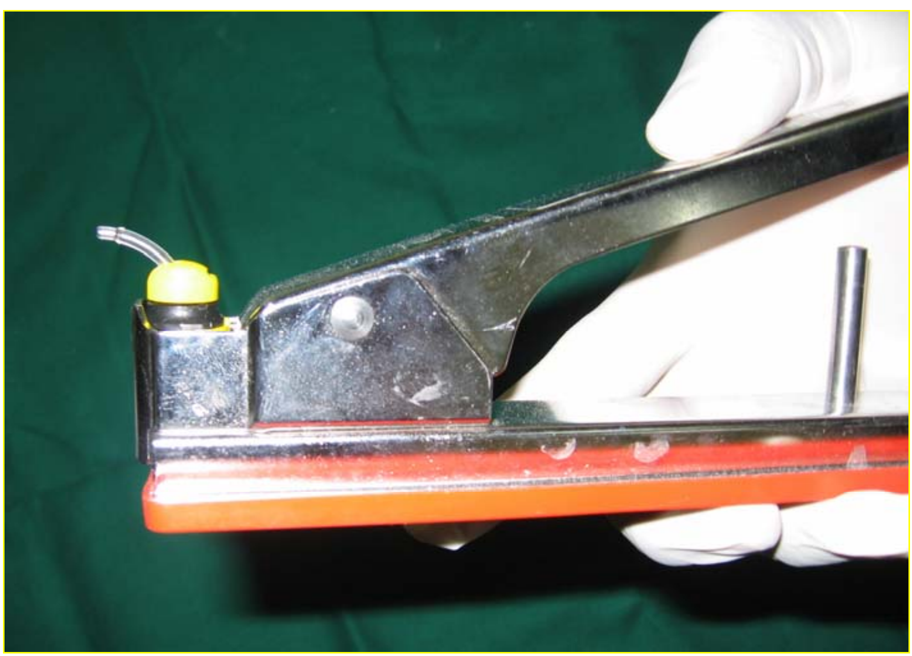


Fig.38 RelyX Unicem capsule inserted into Applicap activator

Fig.38A RelyX Unicem capsule mixed in high frequency mixing amalgamator

Fig.39 RelyX Unicem capsule attached to applicator

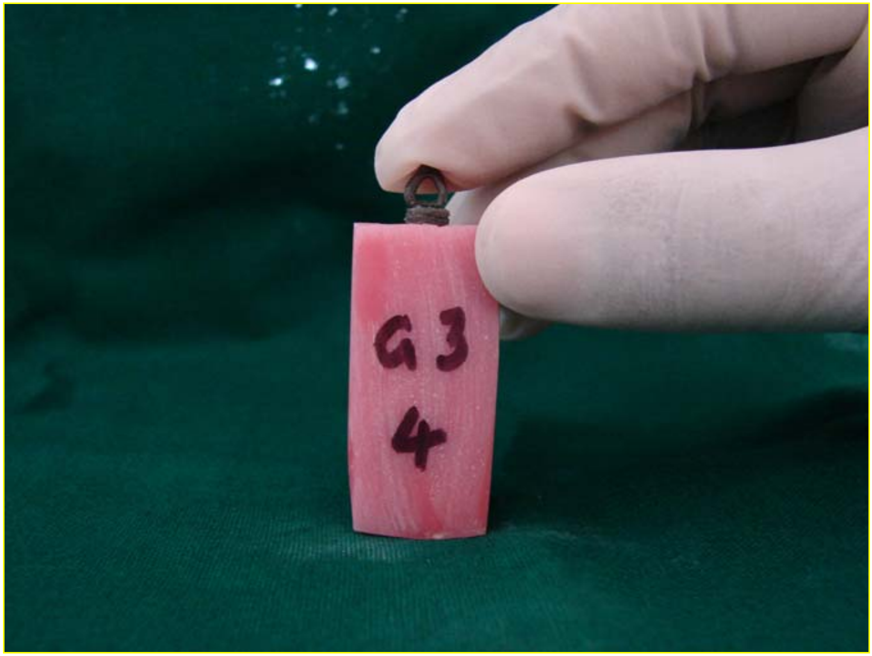


Fig.39A Cementation of metal disc to the tooth specimen

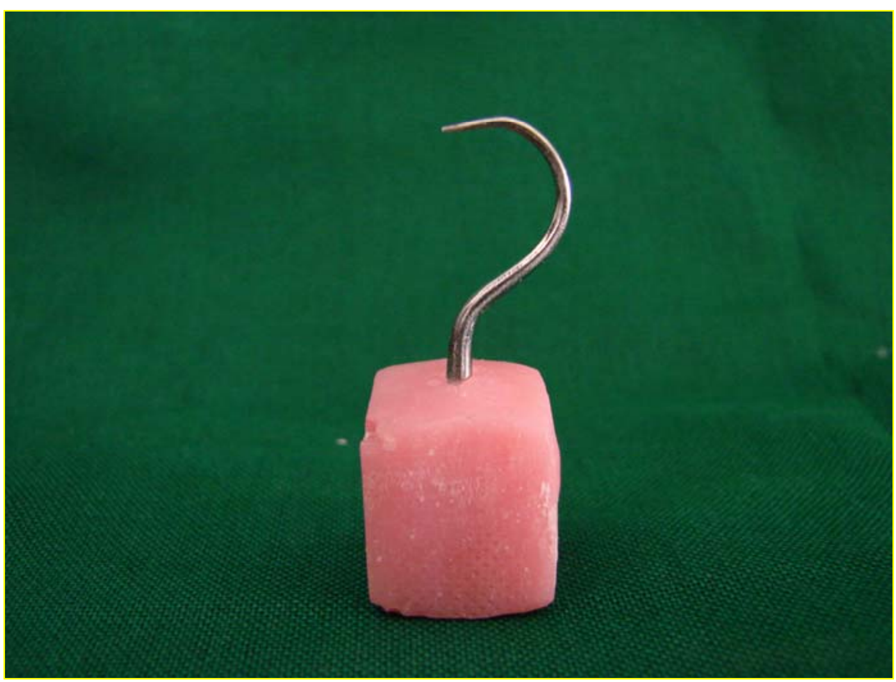


Fig.40 Custom made hook for tensile testing

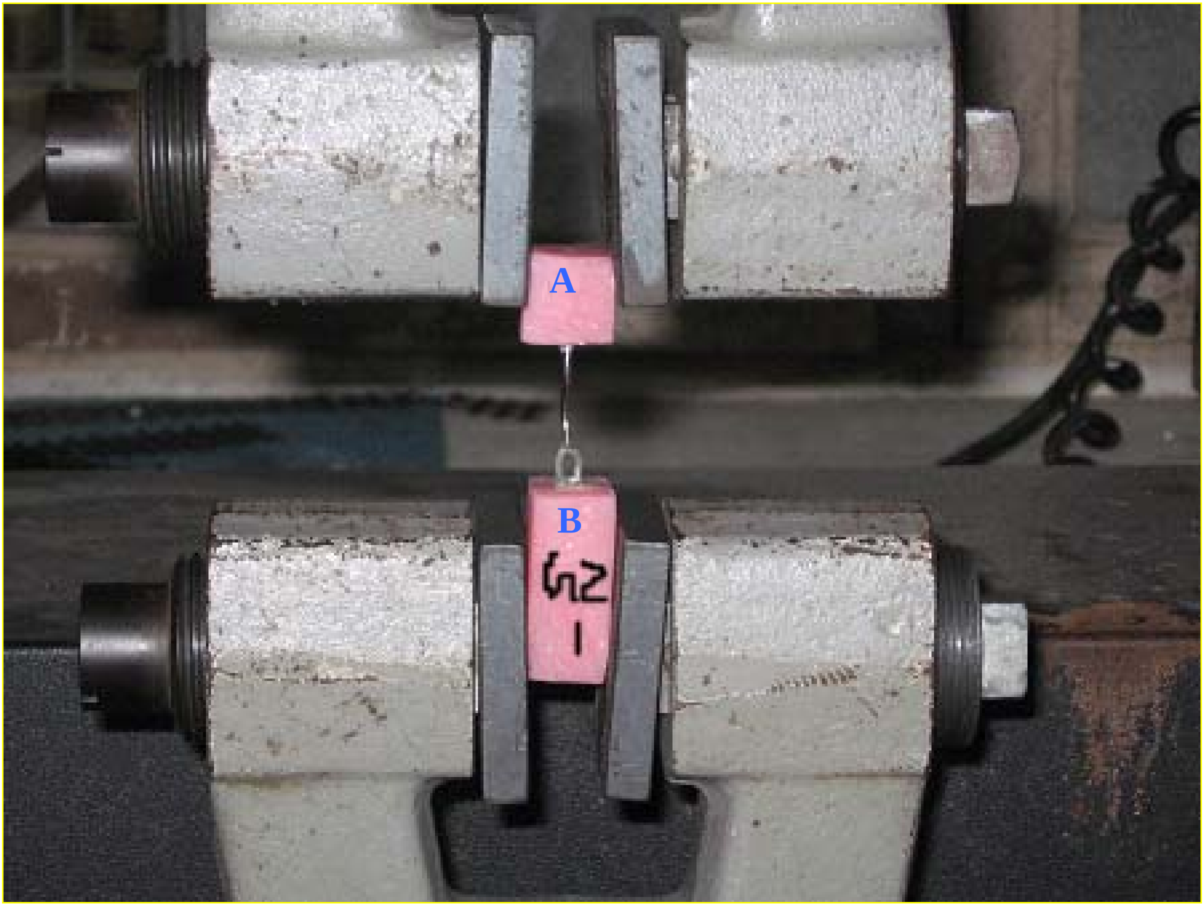


Fig.41 Samples loaded in Instron testing machine
A. Custom made hook engaging the specimen
B. Test specimen

SEM PICTURES

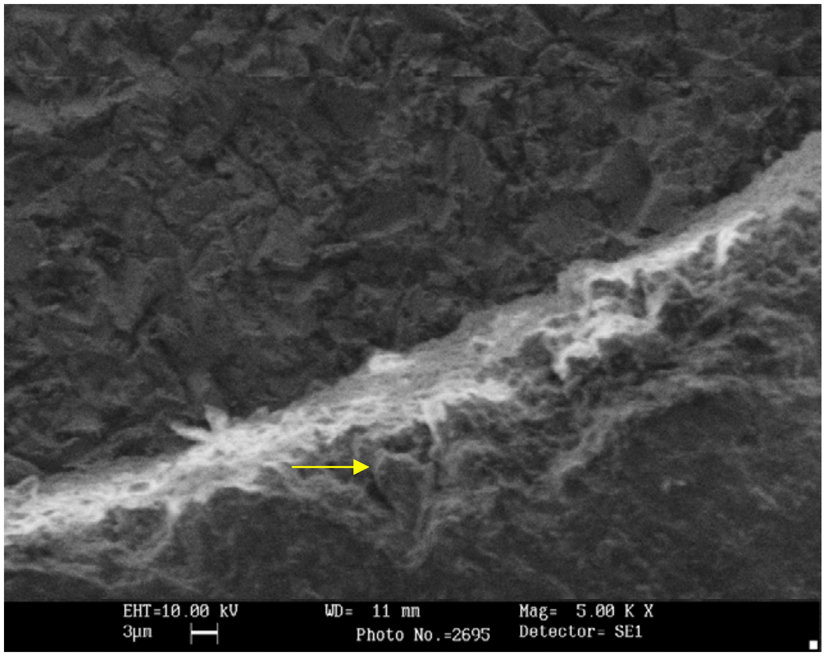


Fig 42 SEM picture of group 1 sample
Arrow indicates cracks formed in the cement surface

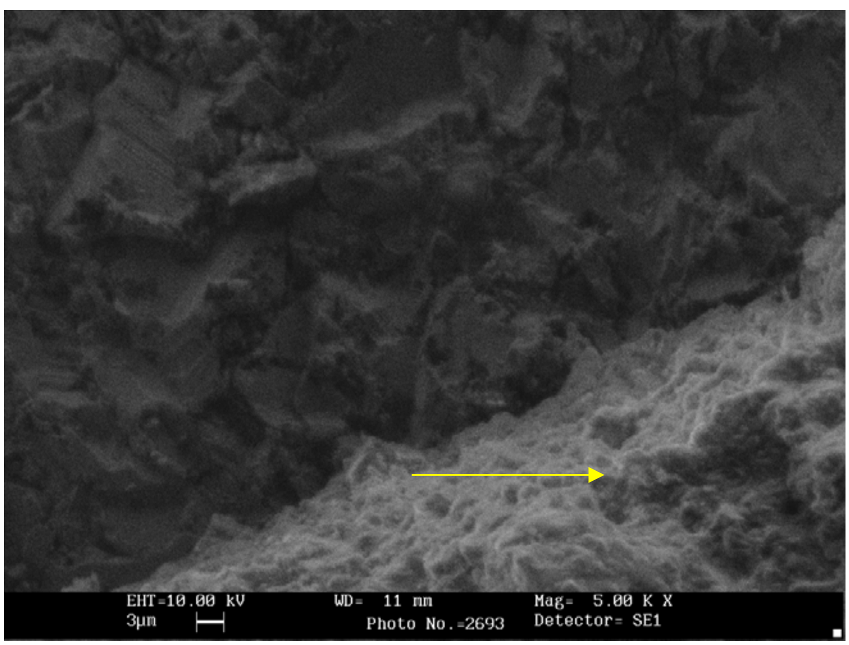


Fig 43 SEM picture of group 3 sample
Arrow indicates cracks formed in the cement surface

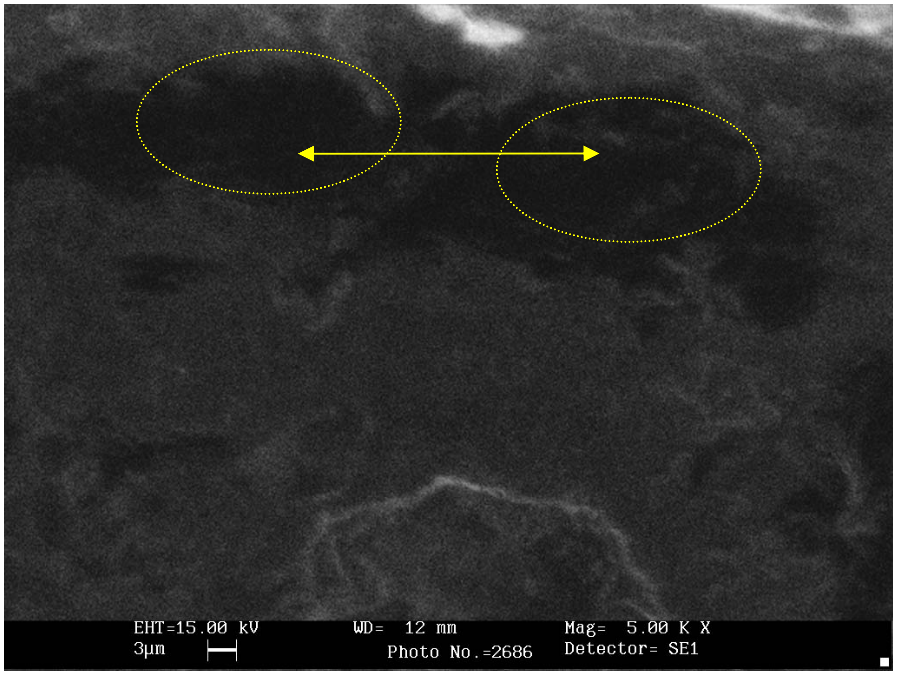


Fig 44 SEM picture of group 2 sample
Arrow indicates debonding of cement from the metal surface

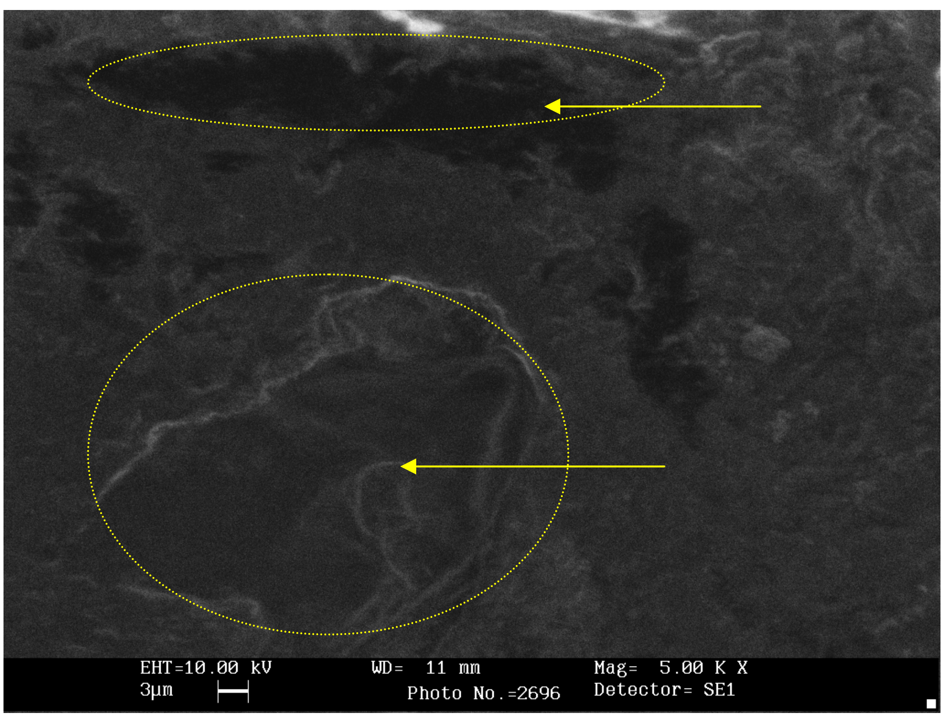


Fig 45 SEM picture of group 4 sample
Arrows indicates debonding of cement from the metal surface

Results

Results

The following results were drawn from the study:

Tables 1, 2, **3,** and **4** show the basic and mean values of the tensile bond strengths obtained from the study for groups **1**, **2**, **3**, and **4** in Mega Pascal.

Graphs 1, 2, 3 and **4** show the basic and mean values of the tensile bond strengths obtained from the study for groups **1**, **2**, **3**, and **4** in Mega Pascal.

The above results were subjected to statistical analysis:

- The SPSS software package was used for statistical analysis (SPSS for Windows 8.0, SPSS Software Copr, Munich, Germany).
- Mean and standard deviation were estimated from the sample for each study group.
- Mean values were compared by student's t-test
- Equality of variances was studied by Levene's test
- In the present study, $p < 0.05$ was considered as the level of significance.

Table 5 Test for significance for comparison of tensile bond strength of two different resin cements used to bond base metal alloy to human enamel.

Table 6 Test for significance for tensile bond strength of two different resin cements used to bond base metal alloy to human enamel with 50 µm aluminum oxide surface treatment on the alloy.

Table 7 Test for significance for tensile bond strength of two different resin cements used to bond base metal alloy to human enamel with 250 μm aluminum oxide surface treatment on the alloy.

Table 8 Test of significance for tensile bond strength of two different resin cements bonded to base metal alloy to human enamel with different grades of air abrasive as surface treatment.

Graph 5 Test for significance for comparison of tensile bond strength of two different resin cements used to bond base metal alloy to human enamel.

Graph 6 Test for significance for tensile bond strength of two different resin cements used to bond base metal alloy to human enamel with 50 μm aluminum oxide surface treatment on the alloy.

Graph 7 Test for significance for tensile bond strength of two different resin cements used to bond base metal alloy to human enamel with 250 μm aluminum oxide surface treatment on the alloy.

Graph 8 Test of significance for tensile bond strength of two different resin cements bonded to base metal alloy to human enamel with different grades of air abrasive as surface treatment.

Table 1:

Tensile bond strengths of Panavia resin cement used to bond base metal alloy to human enamel after surface treatment of alloy with 50μm aluminum oxide.

Sample No	Tensile bond strength in MPa
1	11.57
2	10.23
3	14.32
4	15.12
5	12.32
6	14.11
7	16.22
8	9.89
9	10.56
10	12.02
Mean	12.67

Table 2:

Tensile bond strengths of Panavia resin cement used to bond base metal alloy to human enamel after surface treatment of alloy with 250μm aluminum oxide.

Sample No	Tensile bond strength in MPa
1	10.12
2	9.86
3	11.22
4	12.15
5	13.22
6	9.54
7	8.9
8	12.13
9	10.12
10	13.56
Mean	11.08

Table 3:

Tensile bond strengths of RelyX Unicem resin cement used to bond base metal alloy to human enamel after surface treatment of alloy with 50μm aluminum oxide.

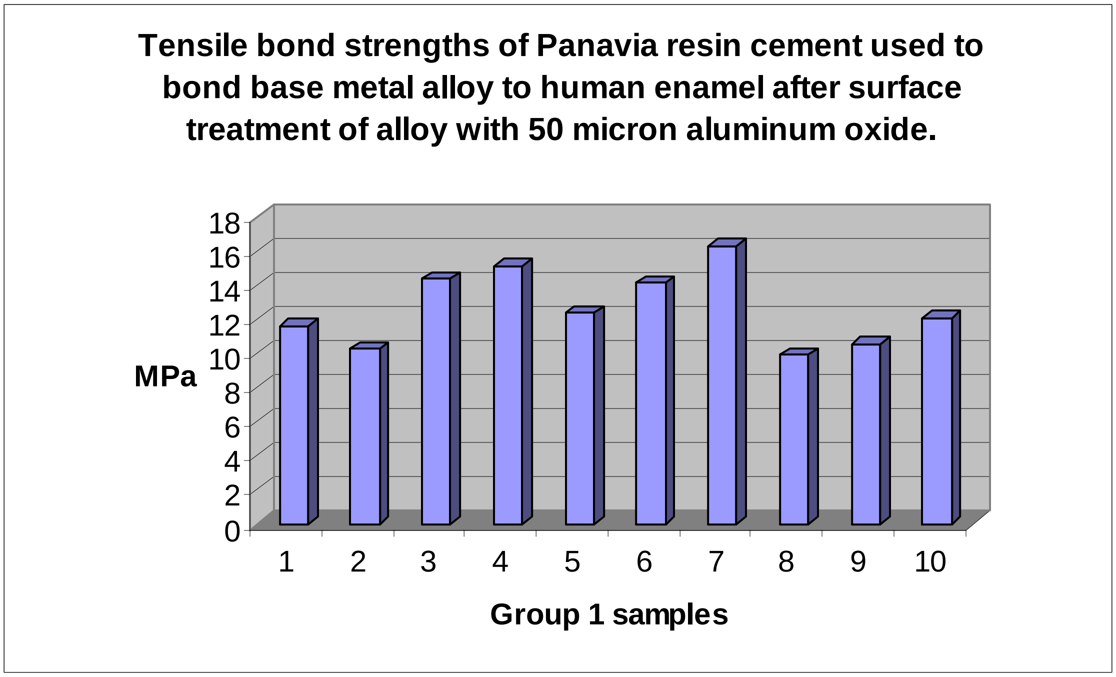
Sample No	**Tensile bond strength in MPa**
1	11.22
2	12.44
3	14.56
4	10.12
5	11.42
6	14.32
7	13.12
8	12.15
9	11.62
10	10.59
Mean	12.16

Table 4:

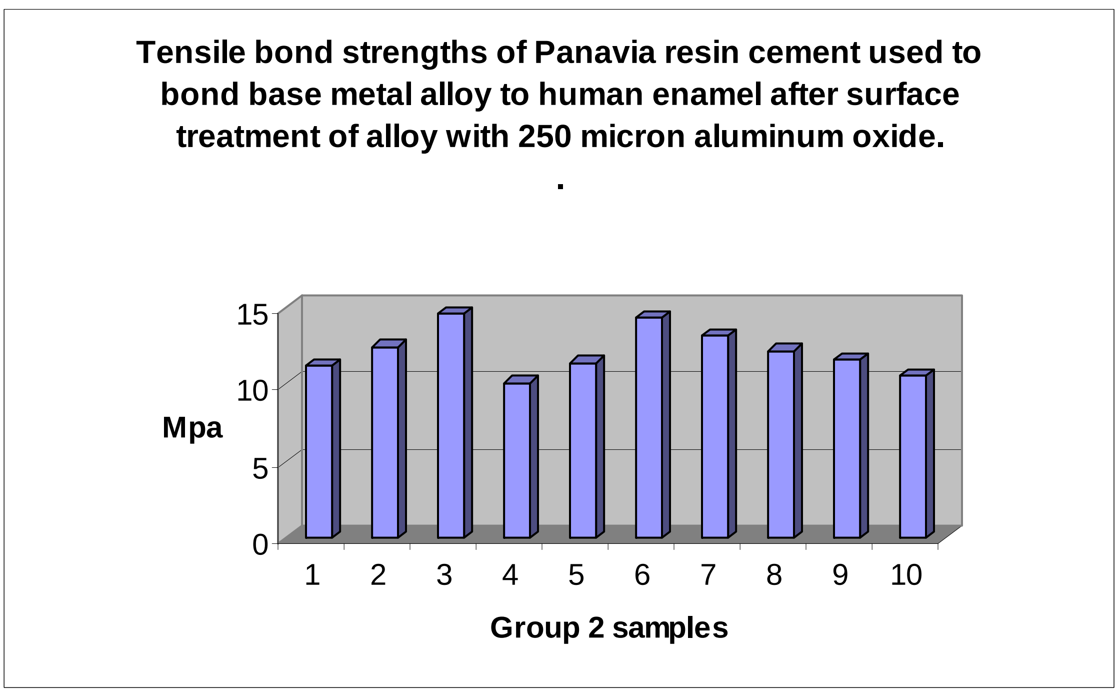
Tensile bond strengths of RelyX Unicem resin cement used to bond base metal alloy to human enamel after surface treatment of alloy with 250μm aluminum oxide.

Sample No	**Tensile bond strength in MPa**
1	10.12
2	11.1
3	10.26
4	11.42
5	9.16
6	12.52
7	10.32
8	9.78
9	10.59
10	12.34
Mean	10.71

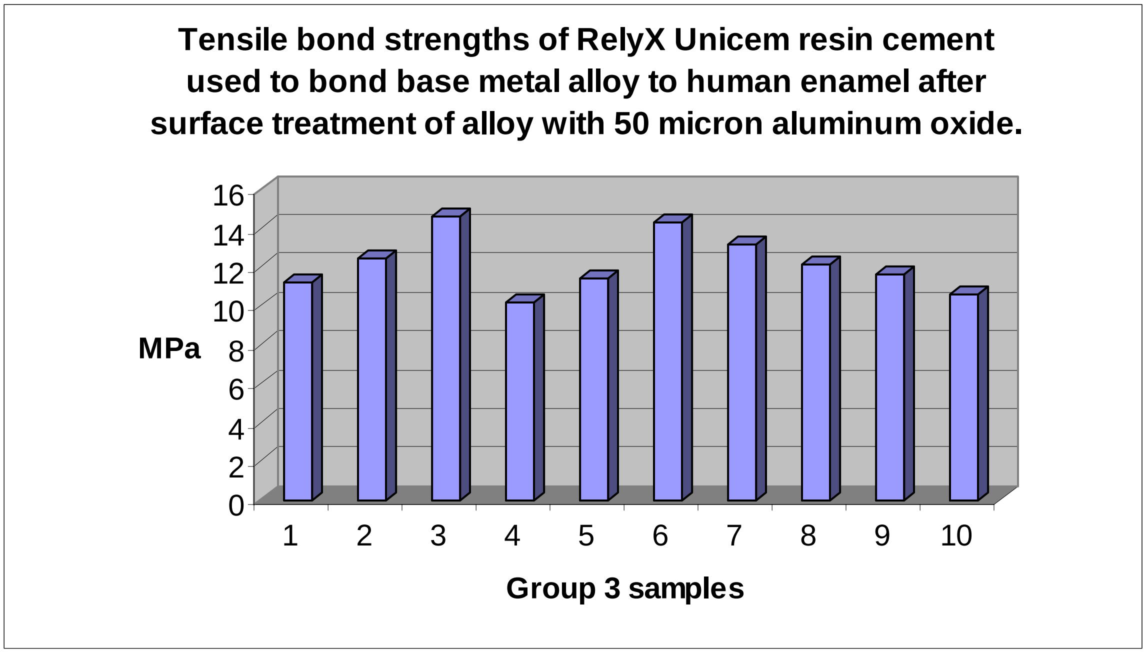
GRAPH 1:



GRAPH 2:



GRAPH 3:



GRAPH 4:

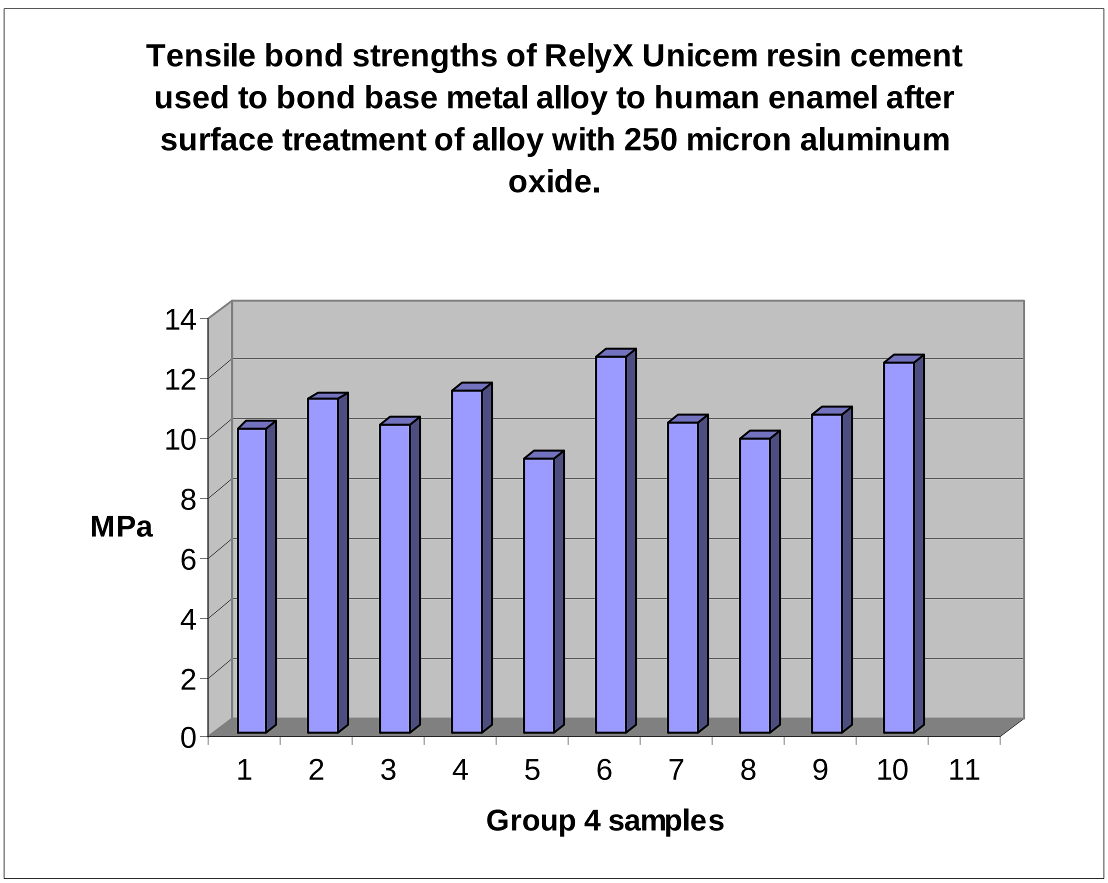


Table 5:

Test for significance for comparison of tensile bond strength of two different resin cements used to bond base metal alloy to human enamel.

RESIN CEMENT SYSTEM	NUMBER OF SAMPLES	MEAN (MPa)	STANDARD DEVIATION	p- VALUE
Panavia F.2	20 (Group 1&2)	11.8590	2.037	0.478
Rely X Unicem	20 (Group 3&4)	11.4585	1.452	

INFERENCE:

The mean tensile bond strength of the two resin cements was 11.86 MPa for Panavia F.2 (Group 1&2) and 11.46 Mpa for Rely X Unicem (Group 3&4) The mean bond tensile bond strength was found to be statistically insignificant with both the cement systems.

GRAPH 5:

Test for significance for comparison of tensile bond strength of two different resin cements used to bond base metal alloy to human enamel.

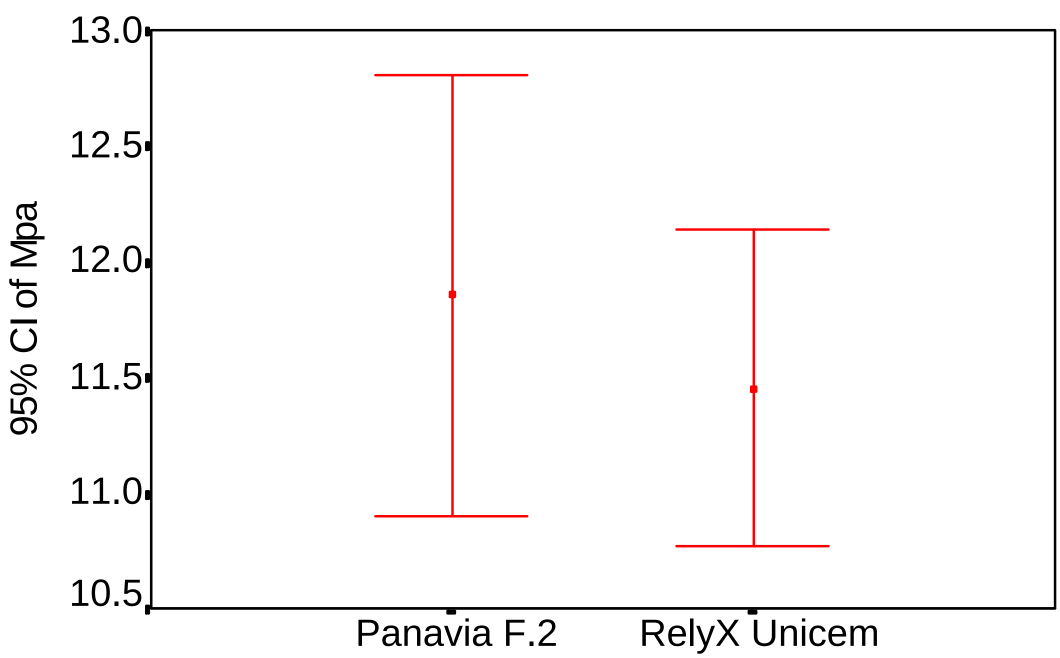


Table 6:

Test for significance for tensile bond strength of two different resin cements used to bond base metal alloy to human enamel with 50 μm aluminum oxide surface treatment on the alloy.

RESIN CEMENT SYSTEM	NUMBER OF SAMPLES	MEAN (MPa)	STANDARD DEVIATION	p-VALUE
Panavia F.2	10(Group 1)	12.6360	2.193	0.117
Rely X Unicem	10(Group 3)	12.1560	1.4822	

INFERENCE:

The mean tensile bond strength of the two resin cements was 12.64 MPa for the Panavia F.2 resin cement (group 1) and 12.15 Mpa Rely X Unicem (group 3) resin cement system. The mean tensile bond strength was found to be statistically insignificant with 50μm aluminum oxide surface treatment on the base metal alloy surface.

GRAPH **6:**

Test for significance for tensile bond strength of two different resin cements used to bond base metal alloy to human enamel with 50 μm aluminum oxide surface treatment on the alloy.

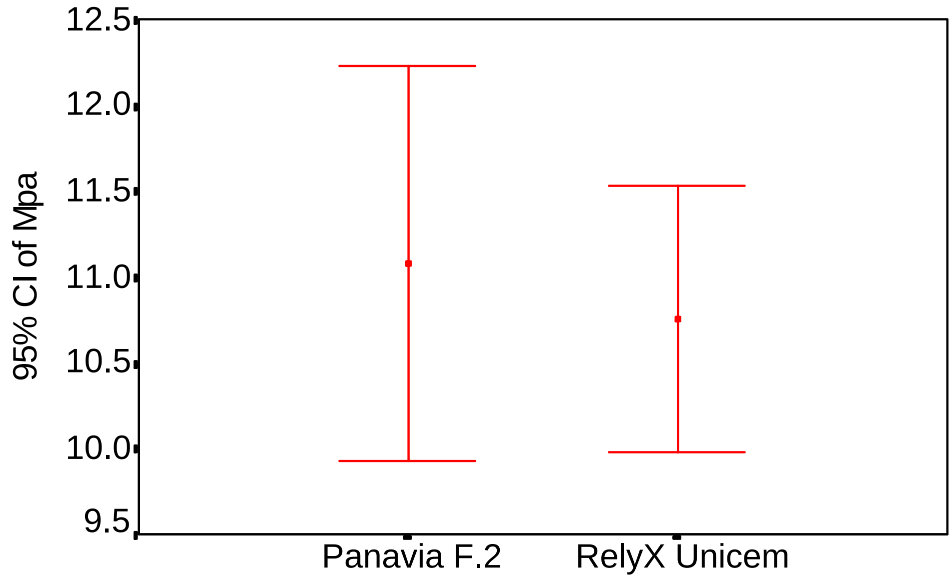


Table 7:

Test for significance for tensile bond strength of two different resin cements used to bond base metal alloy to human enamel with 250 μm aluminum oxide surface treatment on the alloy.

RESIN CEMENT SYSTEM	NUMBER OF SAMPLES	MEAN (MPa)	STANDARD DEVIATION	P VALUE
Panavia F.2	10(Group 2)	11.082	1.614	0.098
Rely X Unicem	10(Group 4)	10.761	1.083	

INFERENCE:

The mean tensile bond strength of the two resin cements was 11.08 MPa for the Panavia F.2 (group2) resin cement system and 10.76 Mpa for the Rely X Unicem resin (group4) cement system. The mean bond tensile bond strength was found to be statistically insignificant with 250μm aluminum oxide surface treatment on the base metal alloy surface.

GRAPH 7

Test for significance for tensile bond strength of two different resin cements used to bond base metal alloy to human enamel with 250 μm aluminum oxide surface treatment on the alloy.

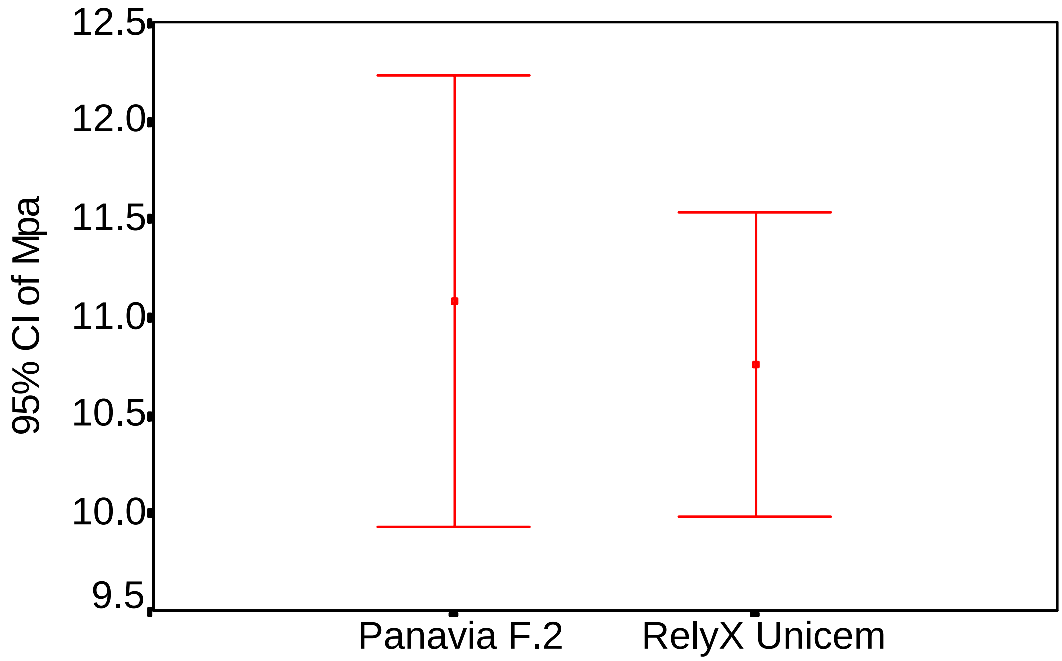


Table 8:

Test of significance for tensile bond strength of two different resin cements bonded to base metal alloy to human enamel with different grades of air abrasive as surface treatment.

ALUMINUM OXIDE ABRASIVE PARTICLE SIZE IN MICRONS	NUMBER OF SAMPLES	MEAN (MPa)	STANDARD DEVIATION	p- VALUE
50	20 (Group 1&3)	12.3960	2.037	0.006**
250	20 (Group 2&4)	10.9215	1.452	

NOTE:

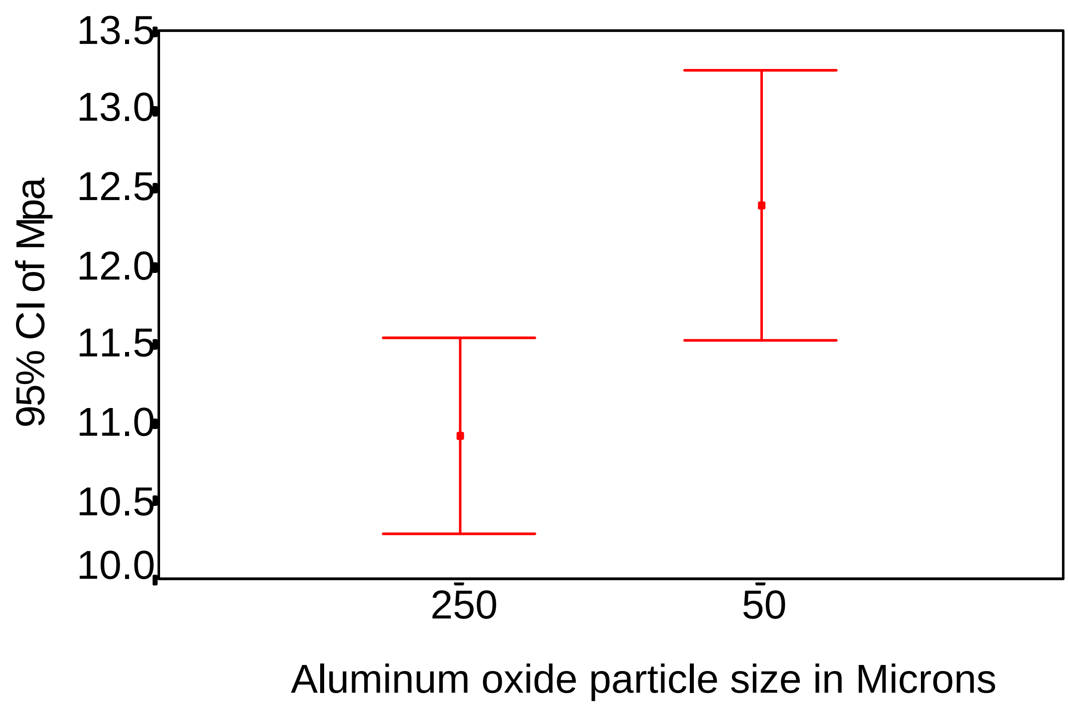
** Denotes significant at 1% level

INFERENCE:

The mean tensile bond strength of two resin cement systems obtained by surface treatment of air abrasion by aluminum oxide of particle size 50µm was 12.40 Mpa and 10.92 Mpa for 250µm. The effect of surface treatment of different grades of aluminum oxide air abrasion on mean tensile bond strength of two different resin cements was statistically significant with a p-value of 0.006 .

GRAPH 8:

Test of significance for tensile bond strength of two different resin cements bonded to base metal alloy to human enamel with different grades of air abrasive as surface treatment.



Discussion

Discussion

Luting cements must withstand masticatory and parafunctional stresses for many years in a warm and wet oral environment. They must maintain their integrity while transferring stresses from crowns or fixed partial dentures to tooth structure. Stress causes deformation, which can range from recoverable elastic deformation, the permanent plastic deformation, and to a fracture[37].

The actual masticatory and parafunctional stresses imposed on luting cements may be much greater than those estimated by simply dividing applied masticatory loads by the resisting surface area of tooth preparations[44,63,45]. In vitro modeling techniques have demonstrated that high stresses are imposed on luting cements, particularly in the biologically important marginal areas[44,63]. Such localized stress concentrations are probably the initial sites of cement failure. Data from an in vivo microleakage study was more consistent with an "all or none" cement failure by micro fracture than with a gradual failure by dissolution[23,45]. Micro fracture may lead to micro leakage, ingress of bacteria and hence caries, or restoration dislodgement.

While using a traditional non-adhesive luting agent such as zinc phosphate, retention is dependent on the geometric form of the tooth preparation that limits the paths of displacement of the cast restoration [53]. In practice, ideal axial wall convergence is rarely obtained[45], and lack of retention is a common cause of fixed prosthesis failure. A reliable adhesive luting agent would therefore enhance the retention of prosthesis. The effect of luting agents on casting retention has been assessed with in-vitro studies[58].

Adhesive resin luting agents have popularized conservative restoration such as porcelain inlays, veneers and resin-retained fixed partial dentures. The resin retained fixed partial dentures in particular, demand a reliable bonding system, though many authors also recommend some mechanical tooth preparation to provide resistance form rather than relying exclusively on adhesion. Laboratory studies have examined the bond strength of various luting agents used for resin-retained prosthesis[21,13,2,7]. These studies have also studied the effect of conditioning of the metal surface and the enamel surface before luting on the bond strength of the luting agents. The surface treatment such as air abrasion, acid etching, tin plating were done on the alloy surface, where as etching, and intra oral air abrasion done on the enamel surface. The summarized results of some of these comparisons revealed considerable variation in the bond strength.

Clinical experience has led to the use of resinous cements for luting Maryland resin-bonded fixed partial dentures. Maryland fixed partial denture are interesting because they have minimum resistance or retention form. Successful use of resinous cements for Maryland fixed partial dentures may be due to not only to their superior adhesive qualities, but also to their remarkably high toughness, namely, energy absorption. Deformation of resinous luting cements in the marginal areas of “Maryland” resin-bonded fixed partial dentures may have limited biologic consequences due to placement of margins on supra gingival enamel and to good adhesion between resinous cements and enamel or casting alloy[37]. Since Maryland fixed partial dentures requires thin metal retainers because of conservative preparation limited to enamel alone, base metal alloy is the alloy of choice. Hence, in this study the tensile bond strength of two different resin cements used to bond base metal alloy to human enamel were determined and compared.

The enamel-to-resin bond is micromechanical in nature for resin cements. It depends upon proper etching and bonding procedures. Though resin cements have been found to provide optimum retention, they may cause postoperative sensitivity in some cases. In order to reduce the postoperative sensitivity, Christensen G.J[15] has recommended the use of resin cements with an acceptable bonding agent. He also suggested that the most popular brand of resin cement for routine use, were a resin system in which a self-etched primer is used for conditioning the tooth surface followed by application of a dual cure cement (Panavia F.2) and a newly introduced self-adhesive universal resin cement (Rely X Unicem) in which self-etching primer was incorporated in the cement itself, and thus separate conditioning of tooth surface was not necessary. Piwowarczyk A[49]et al studied the shear bond strength of cementing materials and concluded that the self-adhesive universal cement RelyX Unicem had higher bond strength values. However in the literature, studies comparing the tensile bond strength between the resin cement system in which the self-etched primer is not incorporated into the dual cure system (Panavia F.2) and self-etched primer incorporated in the dual cure cement system (Rely X Unicem) are less. Hence they were chosen as the test materials in this study.

Various surface treatments, including surface roughening, chemical, and electrolytic etching, can be used to improve the bond strengths of casting alloys. However, there are several unresolved problems with these techniques, for eg.,the etching devices are expensive, they are technique-sensitive, and some base metal alloys are resistant to electrolytic etching[28]. Sandblasting with aluminum oxide is less technique-sensitive than electrolytic etching in producing a roughened alloy surface. The mechanical removal of the debris can also improve wetting before the application of adhesives. This method is inexpensive and eliminates numerous problems associated with etching[28].

Musil and Tiller[54] had suggested that by sandblasting the base metal alloy surface could increase the adhesion. Sen et al[54] did surface treatment with 250µm of aluminum oxide and obtained good results of bond strength for base metal alloys compared to noble and high noble alloys for bonding with resin cements. Fifty microns of aluminum oxide air abrasive has been recommended by the resin cement manufacturer for proper bonding. Hence, the effect of surface treatment of alloy with aluminum oxide of particle size 50µm and 250µm was also investigated in the study.

Literature indicates various laboratory studies have been done on layer thickness[3], adherence energy[6], polymerization shrinkage[60], and the effect of storage conditions of cast restoration[1] on the bond strength of resin cements. The bond strength between the resin luting agent and the dental structure is an important feature that must be investigated. Comparisons among different studies are complicated because of the different approaches used to test adhesive ability (bonding). Generally, adhesive capacity has been evaluated with invitro testing, with shear and tensile tests. However, a study using finite element analysis[10] concluded that shear test were the most efficient to disclose the cohesive resistance of the material, whereas tensile tests were better to investigate the adhesion at the interface. Since the purpose of this study was to evaluate the adhesive capacity of the resin cement rather than the stress produced during clinical function, a tensile test was used. This study was conducted to compare tensile bond strength of two different resin cements used to bond the base metal alloy to human enamel.

In this present study, freshly extracted, caries-free human incisors were used. Incisors were preferred in this study because flat enamel surface could be prepared, which would give a wider area of enamel to be treated and bonded to resin. The selected teeth were mounted in a chemically-cured acrylic resin block, such that it would facilitate the placement of specimen at one end of Instron Universal testing machine without producing any deleterious effects on the tooth specimens.

In order to standardize the dimensions of the metal disc, custom-made metal block with circular wells was used to obtain standardized pattern resin discs. Relining the pattern resin discs on the tooth surface improved their adaptation. The sprues were attached, invested and casting procedure was carried out. In this way, 40 test specimens of corresponding natural tooth and metal discs were obtained. These discs were randomly divided into 4 groups of 10 samples in each group. Group 1 and Group 2 metal discs were surface treated with 50μm and 250μm aluminum oxide respectively and then bonded to enamel of the corresponding natural tooth test specimen with a dual cure resin cement system (Panavia F2.0) in which a self-etch primer was used for conditioning the tooth surface before bonding, according to manufacturer's instructions. Group 3 and Group 4 metal discs were surface treated with 50μm and 250μm aluminum oxide respectively and then bonded to enamel of the corresponding natural tooth test specimen with a dual cure resin cement system (Rely X Unicem) according to manufacturer's instructions. In this resin cement system, the self-etch primer was incorporated into the cement itself and there was no need for separate conditioning of the enamel.

Thermocycling in vitro is a common way of testing dental material to aid in establishing suitability for in vivo use. Hence, in this study, all the 40 test specimens were subjected to thermocycling. Nelsen et al[47] measured temperature extremes at the inner tooth surface below a resin restoration and reported a range of 9° to 52° C. So in this study, all the test specimens were thermocycled between 8° C and 55°C in water bath for 500 cycles with a 20 sec dwell time and 10 seconds transfer time.

The results of this study after tensile testing have been tabulated as a basic data and interpretation of this data was done by statistical analysis.

The basic data (Table no 1) shows a mean value of 12.67 MPa of tensile strength for PanaviaF.2 resin cement with 50μm air abrasion on the alloy surface (Group1). The Table no 2 shows a mean value of 11.08 MPa for Panavia resin cement with 250µm air abrasion on the alloy surface (Group2). The Table no 3 shows a mean value of 12.16 MPa for RelyX Unicem resin cement with 50µm air abrasion on the alloy surface (Group3). The Table no 4 shows a mean value of and 10.71 MPa for RelyX Unicem resin cement with 250µm air abrasion on the alloy surface (Group4). The statistical analysis by Levene's test for equality of variance and Student t test, indicated that the difference in the tensile bond strength for the two different resin cements, showed the p value of >0.478(Table 5). This denotes that the difference in tensile bond strengths between the two test resin cement systems in this study was statistically insignificant.

The bond strength values ranged from 8.9 to 16.22 MPa for the test cements as was inferred from the Tables 1 to 4. A wide range of enamel-resin-composite-metal bond strength has been reported in the literature [19,2,7]. The results from this study are within the range, reported in the literature. These variations are probably due to the standardization difficulties of enamel component in enamel- composite resin -metal joints[19,2,7]. Previous studies by Cotert HS et al[20] and Tjan AH et al[58] indicated that the differences among the bond strengths of various resin composites were statistically significant. In this study, however, no statistically significant difference between the tensile bond strength of dual cure resin cement system (Panavia F.2) in which self-etching primer was supplied separately and a dual cure resin cement system (RelyX Unicem) in which the self-etching primer is incorporated within the cement itself was observed. The differences in bond strengths of individual test specimens were probably due to the relative affinity of the different adhesive groups within the cements to the sandblasted alloy surface as suggested by Atta et al[7].

In this study, different grades of air abrasion of aluminum oxide produced statistically significant results. It was found that surface treatment with 50μm of air abrasive on the alloy surface produced higher mean tensile bond strengths for both the test cements, as compared to surface treatment with 250μm. These findings are contrary to those obtained by Sen et al[54] in their study who concluded that surface treatment with 250μm yielded good results of bond strength for base metal alloy samples compared to noble and high noble alloys. Coelho et al[19]studied the effect of surface treatment with an abrasive of 50μm aluminum oxide, a combination of 50:50aluminum oxide and glass beads and glass beads alone on nickel chromium alloy on the tensile bond strength of resinous cement and concluded that there was no significant difference in the tensile bond strength among the different abrasives used.

SEM examination was done to examine the type of failure between the alloy and cement interface. The alloy specimen surface treated with 50µm air abrasive for both the cement types showed better adhesion of the cement. In some areas cracks in the cement layer were observed (Fig.42&43). The specimen surface treated with 250µm showed frank areas of alloy exposure where the cement had debonded (Fig.44 & 45). The larger grit size could have produced larger surface alterations and contributed to reduced bond strength values in this study. The increase in tensile bond strength of alloy specimen surface treated with 50µm aluminum oxide can be attributed to the smaller grit size, which would have produced smaller surface irregularities[54]. These results are in concurrence with the manufacturer's suggestion for employing 50µm grit size for air abrasion of the metal surface prior to bonding[48].

The mean tensile bond strengths obtained from this study for the two resin cement systems was statistically insignificant which implies that under the test conditions both the resin cements employed performed similarly.

Studies by Cotert HS et al[20] and Tjan AH et al[58] have reported that the Panavia system shows increased bond strength and increased retention amongst the other resin and other luting cements. However, resin cements are highly technique sensitive and surface preparation of enamel and metal and the bonding protocol can influence the result outcome[19]. Resin cements with incorporated self-etched primer may be of value in saving operator's time and elimination of errors, which can be caused during etching and priming procedures as with conventional resin cements. However, their performance under varied laboratory and clinical conditions needs further investigation.

In this study base metal alloy was employed with air abrasion as the surface treatment for estimating the bond strength of two resin cement systems to enamel. Further investigations regarding the tensile bond strength of these resin cements bonded to different materials such as ceramics, noble alloys, etc and surface treatments other than air abrasion are also needed to enhance the results obtained with this study. Similarly studies performed under clinical conditions and long term clinical data are also required to obtain more predictable conclusions about the performance of these materials.

Conclusion

CONCLUSION

The following conclusions were drawn from the results obtained in this in-vitro study of comparison of tensile bond strength of two different resin cements used to bond base metal alloy to human enamel:

1. The mean tensile bond strength of two resin cement systems used to bond base metal alloy to human enamel after the surface treatment of alloy with 50µm aluminum oxide air abrasive was 12.67MPa for the group 1 samples and 12.15MPa for the group 3 samples. The difference in the mean tensile bond strength was found to be statistically insignificant.
2. The mean tensile bond strength of two resin cement systems used to bond base metal alloy to human enamel after the surface treatment of alloy with 250µm aluminum oxide air abrasive was 11.08MPa for the group 2 samples and 10.71MPa for the group 4 samples. The difference in the mean tensile bond strength was found to be statistically insignificant.
3. The comparative evaluation of the mean tensile bond strength of the resin cement systems used to bond base metal alloy to human enamel was found to be 11.86MPa for the group 1 and 2 samples and 11.46MPa

for the group 3 and 4 samples, which was found to be statistically insignificant.

4. The mean tensile bond strength of two resin cements obtained by surface treatment with air abrasion of 50µm aluminum oxide on the alloy was 12.40MPa and 10.92MPa for 250µm aluminum oxide. The mean tensile bond strength of the two resin cements obtained by surface treatment with air abrasion of 50µm aluminum oxide to the alloy surface was found to be higher than those obtained by surface treatment with air abrasion of 250µm aluminum oxide. This difference was found to be statistically significant.
5. Scanning electron microscope examination of metal and cement interface revealed adhesive type of failure with 250µm aluminum oxide air abrasion, whereas, cracks were found in the cement layer with 50µm aluminum oxide air abrasion irrespective of the resin cement employed.

Summary

SUMMARY

This in-vitro study has been done to evaluate and compare the tensile bond strength of two different resin cements used to bond base metal alloy to human enamel. The two test resin cement systems were Panavia F.2 and Rely X Unicem. A total of 40 natural teeth were taken in this study. The labial surfaces were ground at slow speed and were visually examined to ensure that enamel was continuous and the dentin was not exposed. Forty alloy discs of 5 mm diameter with 1mm thickness were randomly divided into four groups of ten samples each. Group 1 and Group 2 alloy discs were surface treated with 50μm and 250μm aluminum oxide respectively and then bonded to enamel of the corresponding natural tooth test specimen with Panavia F2.0, according to the manufacturer's instructions. Group 3 and Group 4 alloy discs were surface treated with with 50μm and 250μm aluminum oxide respectively and then bonded to enamel of the corresponding natural tooth test specimen with Rely X Unicem according to the manufacturer's instructions.

The samples were thermocycled and they were loaded in Instron testing machine for tensile testing. The debonded alloy surfaces were examined under scanning electron microscope. The results were tabulated and subjected to statistical analysis.

The results of this study showed the comparative mean tensile bond strengths of two resin cements were not statistically significant. The effect of surface treatment with 50 μm aluminum oxide of air abrasion of the base metal alloy surface yielded significantly higher values of tensile bond strength compared to that obtained with 250 μm aluminum oxide of air abrasion in this study. SEM examination of the metal interface revealed adhesive type of failure with 250μm aluminum oxide air abrasion whereas, cracks were found in the cement layer with 50 μm aluminum oxide air abrasion irrespective of the cement employed.

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